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TUNGSTEN SHEET ROLLING PROGRAM

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UNIVERSAL-CYCLOPS SPECIALTY STEEL DIVISION
CYCLOPS CORPORATION

TECHNICAL REPORT AFML-TR-67-311

OCTOBER 1967

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TUNGSTEN SHEET ROLLING PROGRAM

JEROME H. SCHWERTZ

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WILLIAM A. MCNEISH

FOREWORD

This final report covers the work performed under Contract AF 33(600)-41917 from 29 September 1960 to 29 September 1966. The manuscript was released by the author on 15 September 1967 for publication as an AFML technical report.

This contract with the Refractomet Division, Universal-Cyclops Steel Corporation, Bridgeville, Pennsylvania since changed to Universal-Cyclops Specialty Steel Division, Cyclops Corporation, Bridgeville, Pennsylvania, was initiated under Manufacturing Methods Project 7-827, "Tungsten Sheet Rolling Program". It was administered under the technical direction of Messrs. Hugh L. Black and George M. Glenn of the Metallurgical Processing Branch, MATB, Manufacturing Technology Division, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio.

The investigations were originally conducted by Mr. W. J. Schoenfeld, Metallurgical Engineer, and completed by Mr. J. H. Schwertz, Metallurgical Engineer, with Mr. C. P. Mueller and Mr. L. M. Bianchi as Project Coordinators. Significant contributions to this program were made by L. L. France, F. T. Snyder, and A. J. Bandola.

The State-of-the-Art Survey for this contract was conducted by Battelle Memorial Institute under the direction of Messrs. D. J. Maykuth, V. D. Barth, and H. R. Odgen.

Initial extrusion work on the contract was carried out by the TAPCO Division of TRW, Inc., and the extrusion of larger billets was accomplished on DuPont's 2750 ton press located in Baltimore, Maryland. The authors wish to express their appreciation for the cooperation extended.

Since the nature of this work is of interest to many fields of endeavor, any comments are solicited as to the potential utilization of the material produced under this contract. In this manner, it is felt that a full realization of the material produced will be accomplished.

This project has been accomplished as a part of the Air Force Manufacturing Methods Program. The primary objective of the Air Force Manufacturing Methods Program is to develop, on a timely basis, the manufacturing processes, techniques and equipment for use in economic production of USAF materials and components. This program encompasses the following technical areas:

FOREWORD (cont.)

Metallurgy	- Rolling, Forging, Extruding, Drawing, Casting, Powder Metallurgy, Composites.
Chemical	- Propellant, Coating, Ceramic, Graphite, Nonmetallics.
Fabrication	- Forming, Material Removal, Joining, Components.
Electronics	- Solid State, Materials and Special Techniques, Thermionics.

Suggestions concerning additional Manufacturing Methods development required on this or other subjects will be appreciated.

This technical report has been reviewed and is approved.

C. H. Nelson

CHARLES H. NELSON, Assistant Chief
Manufacturing Technology Division
Air Force Materials Laboratory

ABSTRACT

A manufacturing process has been developed to produce unalloyed arc-cast tungsten sheet materials. The processing parameters for optimum physical and mechanical properties were investigated from the raw material to the final product. The investigation included powder consolidation to electrode, ingot melting variables, primary breakdown by extrusion and forging, and a study of the effect of rolling variables on final sheet properties. A scale-up of processing to produce pilot production quantities of 0.020", 0.040", and 0.060" gauge sheet was accomplished, but severe processing and handling problems in all phases of the scale-up, because of the inherent brittleness of the material, prevented the realization of the goal, 36" wide by 96" long sheet. Satisfactory tungsten ingots can be melted with consistent quality up to 6" round diameter and larger ingots up to 9-1/2" round can be melted providing adequate power and cooling capacity are available. Direct forging to sheet bar is not practical and press forging of extruded rounds is not practical due to yield loss on conditioning and an extra operation. Satisfactory extrusion of sheet bar and rounds from conditioned billet diameters up to 6" can be accomplished at extrusion ratios of 4.1:1 and 3000° to 3500°F furnace temperatures. Bend transition results indicate a slight advantage with the extruded sheet bar over press forged bars. The optimum bend transition properties were obtained from material having a minimum 92% reduction from the last recrystallization anneal. Stress relief to improve bend transition must be accomplished at temperatures below the temperature of initial recrystallization. The lowest longitudinal bend transition temperature achieved was 200°F.

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I. Introduction

A. General

By virtue of its high melting point and relative abundance, tungsten occupies a preeminent position among refractory metals for applications in air frames, re-entry vehicles and nozzles and vanes for rocket motors. Much progress has been made in developing massive tungsten parts that suit some of these applications. However, production applications for tungsten in thin sections have been hampered by the limited availability of sheet in the sizes and quality desired for advanced military aircraft.

In recognition of this, the United States Air Force awarded Universal-Cyclops Specialty Steel Division Contract No. AF 33(600)-41917 for the "Development of New or Improved Techniques for the Production of Tungsten Sheet". The purpose of this contract was the establishment of the state-of-the-art of rolling tungsten and tungsten alloy sheet in order to advance the industry capability to economically produce sheet to the required quality and sizes. The overall objective was to develop new and/or improved techniques for rolling large size tungsten sheets. The objective was to be the production of acceptable tungsten sheet 36" x 96" x thickness of .020", .040", and .063" in a flat condition with uniform properties.

In order to accomplish the above aims, the program was broken down into five phases which are summarized below:

1. Phase I - State-of-the-Art Analysis

The objective of this phase was to evaluate the current state-of-the-art of tungsten sheet rolling throughout the sheet mill rolling industry. Also, to plan a detailed program to satisfactorily accomplish the development effort required to advance the state-of-the-art.

2. Phase II - Ingot Process Development

The refinement of tungsten ingot production processes including the establishment of tests and testing procedures to insure satisfactory uniformity of tungsten ingots.

3. Phase III - Development of Rolling Operations

This phase covered the breakdown of tungsten ingots to establish process parameters including analysis of the processing variables. Further, the establishment of processing controls and test procedures for the controlled rolling of tungsten sheet.

4. Phase IV - Process Uniformity Verification and Post Rolling Development

This phase encompassed the controlled rolling of tungsten sheet using processes developed in Phase III. In addition, post rolling development was accomplished in an effort to establish control specifications for the pilot production run.

5. Phase V - Final Pilot Production

The production of sheets 36" x 96" x thicknesses of .020", .040", and .063" was undertaken. The production was designed to demonstrate the reliability of the development process and verification of uniformity of flat sheets produced using the developed techniques.

B. State-of-the-Art Analysis

The state-of-the-art survey was conducted by Battelle Memorial Institute as a sub-contractor to Universal-Cyclops on the above contract. Primary responsibility for planning and conducting this survey w[illegible]tered in the non-ferrous metallurgy division under the direction of H. R. Ogden, Division Chief, with D. J. Maykuth, Assistant Division Chief, and V. D. Barth, Principle Metal-

lurgical Engineer in the Powder Metallurgy Division assisting in this effort. The objectives of this survey were to assist the current state-of-the-art in the rolling of tungsten sheet and to recommend the composition of a tungsten sheet material of materials for evaluation in the Phase II effort.

In conducting this survey use was made of a questionaire, personal interviews, as well as an extensive search of the literature in the Defense Metals Information Center. A summary of this survey is given in Appendix I to this report.

C. Procedure Justification

The results of the state-of-the-art survey indicated that there was insufficient data established on alloys of arc-cast tungsten to warrant a major investigation toward the production of sheet. Since the tungsten sheet rolling program issued to Fansteel Metallurgical Corporation under the Bureau of Naval Weapons Contract NOw 60-0621-c has thoroughly investigated the powder metallurgy approach to the consolidation and fabrication of sheet from unalloyed tungsten and variously doped tungsten powders, the Air Force thought it was needless to duplicate this effort. And finally, to obtain the program objectives of lowest practical temperature transition from ductile to brittle behavior, maximum consistency of grain structure and recrystallization behavior, freedom from lamination and other defects, and chemical homogeneity, arc-melted tungsten was felt to have a much higher probability of success than powder compacted material.

Generally arc-melted tungsten product is characterized by a higher total purity than can be presently obtained by powder metallurgy consolidation practices. The higher purity associated with the arc-melted product may be expected to contribute to greater ductility in this material at elevated temperatures. This has

already been reflected in the successful use of lower rolling temperatures for arc-melted product (after extrusion and forging). By confining the sheet rolling program to arc-cast unalloyed tungsten material, it was felt that the state-of-the-art would be more satisfactorily and rapidly advanced than by having the effort divided between a powder metallurgy approach and an arc-casting approach on pure tungsten and its possible alloys.

To provide the sheet size requirements of the program required ingot sizes considerably larger than those being converted to sheet material. This would introduce new problems in melting and primary breakdown and create the necessity to investigate the effect of numerous processing variables on purity, and mechanical and physical quality of the sheet product. If the production goal of the contract could be accomplished, it would be possible at some future date to readily accomplish sheet production of arc-melted tungsten alloys through suitable laboratory development programs. Therefore, it was recommended and agreed by the Air Force that unalloyed tungsten should be selected as a candidate for arc-melting.

II. Ingot Melting Evaluation

From the state-of-the-art survey, experience in the consumable electrode arc-melting of tungsten accumulated rapidly. At least seven organizations had used this procedure to produce good quality unalloyed tungsten ingots in diameters of 4" or greater. Unalloyed tungsten ingots as large as 9" in diameter had been made. In-house operations at Universal-Cyclops had indicated that actual melting of small developmental tungsten ingots presented no severe problems. Five inch diameter ingots up to 12" long could be made consistently and a standard process had been developed for this operation. Material losses, however, in conditioning small ingots were severe and a 50% to 60% yield was considered normal. The major loss on these ingots was removal of the sidewall rather than top or bottom cropping. By scaling up to larger ingots, the amount of sidewall removed on a relative cross sectional area basis is much less.

The final .063" sheets required in this program weigh approximately 140 pounds and assuming an overall yield of 20% to 30%, ingots weighing approximately 500 pounds are required. Since all wrought forms of tungsten originate from a powder product, tungsten powder was considered as the principle raw material for tungsten sheet. The primary effort in the ingot melting evaluation was to 1) develop specifications for reproducible raw material, 2) standardize electrode preparation techniques, 3) improve yields on ingot sizes presently available, and 4) outline a methodical development for scale-up to larger diameter ingots.

A. Electrode Preparation

1. Raw Material

With all available data, the state-of-the-art survey suggested that the program be based on the utilization of

unalloyed tungsten having no intentional deoxidizing additions. To promote as much uniformity as possible in supplying material for this program, one powder lot of reduced tungsten was reserved for the entire program. Electrodes were to be manufactured in accordance with Universal-Cyclops materials specification WEB 61-3-A given in Appendix II of this report. From this specification, the powder utilized must be produced by the hydrogen reduction of ammonia paratungstate. This method was selected because higher and more consistent purity levels were available by this method of production. The chemistry limits given in this specification are extremely lenient considering the typical electrode chemistries reported; however, the producers required the limits specified to allow for variable powder chemistry and analytical limitations.

From a melting standpoint, the powder particle size does not appear to be critical. However, there are several considerations related to particle size which affect the overall operation. The particle size affects the bulk density which is important in correcting for shrinkage during sintering. The finer powders having lower bulk densities have a faster sintering rate than larger powders and there appears to be almost a straight line relationship between particle size and sintering time-temperature conditions to produce a specified density. From this standpoint, it would be desirable to utilize fine powders. It is also desirable to minimize the degree of shrinkage, especially in producing large electrodes where the pre-sintered electrode is approaching the size limits of press and sintering facilities. Thus a compromise was used in selecting starting particle size. The K-200 type powder selected for this program had a FSSS of 3.35 microns and a resulting bulk density of 68.6 grams per cubic inch. Table I shows percent distribution in each particle size range for the 3.35 micron average particle size powder used throughout this work.

TABLE I

POWDER PARTICLE SIZE DISTRIBUTION
(Measured by Photelometer)

Particle Size (Micron)	% Weight for Micron Range Indicated
0-1	1.84
1-2	10.02
2-3	22.69
3-4	24.51
4-5	17.06
5-6	10.34
6-8	7.35
8-10	3.71
10-12	2.45

The chemistries on this material analyzed by the supplier and by Universal-Cyclops is shown in Table II. As can be noted, the detection limits of the two laboratories are quite different. Also, the values reported by the two laboratories differ considerably on some elements. This is partly due to the fact that the values from the electrode supplier for all elements except carbon, oxygen, hydrogen, and nitrogen are from powder analysis and the Universal-Cyclops analysis is taken from the sintered electrode bar.

Previous evidence on all the refractory metals, including tungsten, indicates that the purity of the starting material affects both the melting conditions and subsequent fabrication. Although very little evidence of this has been documented for tungsten, processing parameters such as extrusion constant, minimum acceptable working temperatures, etc. do show a significant difference based on differences in purity. Minimum interstitial levels are desirable from a fabrication standpoint. The elements most detrimental to fabrication are considered to be iron, silicon, oxygen, and carbon.

From the state-of-the-art survey, intentional carbon additions are used by several melters to promote deoxidation during melting. Although this addition is a prerequisite for molybdenum production, it has been shown that it is not required in tungsten and actually is detrimental in subsequent fabrication. The nominal oxygen content in powder varies from 500 to 1600 ppm. However, this is normally reduced to below 100 ppm during the sintering cycle. In subsequent melting, with no intentional carbon additions, the oxygen content is reduced to as low as 3 to 24 ppm, the average being approximately 10 ppm.

The relatively high and extremely variable oxygen content of the tungsten powder is due primarily to the large sur-

TABLE II

ELECTRODE CHEMISTRY
(% By Weight)

Element	Supplier's Analysis	Universal-Cyclops' Analysis
As	<.0003	<.010
Al	<.00005	<.001
Ca	<.0005	.0003
Co	<.001	.0038
Cr	.0001	.0008
Cu	<.00001	.005
Fe	.0004	.0019
K	<.003	.0013
Mg	<.0003	.002
Mn	<.001	<.001
Mo	.0014	.0085
Na	.0018	.001
Ni	.0002	.0008
Si	<.0003	<.002
Sn	<.001	<.002
C	.001	.0055
O_2	.00053	.0007
N_2	.00023	.0010
H_2	.0002	.0019

<Indicates Limits of Detection

face area of the powder and varies with the particle size. Tungsten powder picks up substantial quantities of oxygen when exposed to air and the amount adsorbed is almost directly proportional to the surface area. From this standpoint, it is desirable to utilize maximum particle size in producing electrode bars.

2. Compaction and Consolidation

For this program all electrode bars were compacted from powder by isostatic pressing as this method has been shown to be the most satisfactory from the standpoint of reproducibility and maximum size availability. There are several basic processing procedures which must be followed in compacting powder into electrodes by this method:

a. In transferring powder to the plastic or rubber container, caution must be exercised to prevent bridging or formation of air pockets.

b. Because the as-pressed bars are very fragile and problems with camber, a length to diameter ratio of 16:1 is considered maximum.

c. The powder must be packed uniformily to permit pressing of straight uniform bars.

Minimum density requirements, outlined by Universal-Cyclops Specification No. WEB 61-3-A in Appendix II, are 90% of theoretical density. It has been found by in-house studies that densities lower than this level are more subject to 1) breakage in handling and 2) difficulties in assembling by both mechanical and welding methods. In assembling, threads are difficult to machine for mechanical joints, and welding causes shrinkage and subsequent cracking due to the low density.

The camber of electrode bars effects to a substantial degree the sidewall condition of the ingot produced. If the electrode has considerable camber, it will be closer to one side of the mold and the opposite side of the ingot will have a poor sidewall. This is also one of the major causes of mold burn through.

Maximum electrode length is desirable to the user because of assembly problems; however, due to the low green strength of the electrodes as-pressed and camber which occurs during sintering, electrode lengths are held at a maximum of 16 times the diameter.

3. Electrode Evaluation

Pressed and sintered electrode bar stock of various diameters were purchased to Universal-Cyclops Specification WEB 61-3-A for the purpose of obtaining optimum electrode to mold ratios for further melting operations. Table III gives the electrode manufacturing data on the various diameters requested. A review of these tables shows the consistency that can be acquired in uniformity of dimensions and shrinkage factors for the various size electrodes by utilizing one powder lot as source material. Figure 1 depicts as-received 1-3/4" diameter pressed and sintered electrodes.

4. Electrode Assembly

Two methods of electrode joining (welding and mechanical) have been studied and will be discussed separately.

a. Welding

Joining of the sintered tungsten electrodes was attempted by the use of TIG welding principles. The bars were loaded onto a rack designed with trunions to facilitate turning of the bar during the welding operation. The ends of the welded area

TABLE III

ELECTRODE MANUFACTURING DATA

Requested Diameter (Inches)	Actual Diameter As-Pressed (Inches)	Actual Diameter Sintered (Inches)	Length As-Pressed (Inches)	Length Sintered (Inches)	Density As-Pressed (%)	Density Sintered (%)	Sintering Shrinkage Diameter (%)	Sintering Shrinkage Length (%)	Max. Deviation From Average Diameter (Inches)		Bar Weight (Lbs)
1.500	1.785	1.540	29.63	24.75	60.2	93.3	13.7	13.6	+0.015	-0.030	30.0
1.500	1.760	1.535	28.69	24.88	60.7	92.3	12.8	13.3	+0.030	-0.035	29.6
1.750	2.090	1.810	20.84	18.06	59.7	91.3	13.4	13.3	+0.050	-0.050	29.6
1.750	2.065	1.765	21.28	18.44	59.6	93.7	14.5	13.3	+0.040	-0.045	29.5
2.000	2.220	1.905	37.63	32.50	57.8	90.5	14.2	13.6	+0.075	-0.025	58.5
2.250	2.630	2.285	25.75	22.41	61.2	93.1	13.1	13.0	+0.070	-0.110	59.6
2.500	2.890	2.500	21.50	18.50	60.3	93.6	13.5	14.0	+0.040	-0.025	59.3
1.750	2.060	1.765	28.10	24.38	57.4	90.1	14.3	13.2	+0.025	-0.015	37.50
1.750	2.055	1.765	28.10	24.19	57.6	90.5	14.1	13.9	+0.020	-0.020	37.55
1.750	2.060	1.760	28.00	24.19	57.3	90.7	14.5	13.6	+0.020	-0.020	37.25
1.750	2.050	1.760	28.10	24.25	57.7	90.5	14.1	13.7	+0.025	-0.020	37.25
1.750	2.050	1.750	28.03	24.19	57.7	91.7	14.6	13.7	+0.015	-0.015	37.20
1.750	2.040	1.755	28.03	24.31	58.3	90.5	14.0	13.3	+0.045	-0.035	37.15
1.750	2.055	1.770	27.91	24.28	57.7	89.0	13.9	13.0	+0.045	-0.030	37.10
1.750	2.045	1.770	27.91	24.31	58.5	89.3	13.4	12.9	+0.015	-0.010	37.20
1.750	2.050	1.770	27.91	24.10	58.0	89.8	13.7	13.7	+0.035	-0.015	37.15
1.750	2.055	1.765	28.10	24.22	57.7	90.5	14.1	13.8	+0.030	-0.025	37.45
1.750	2.055	1.760	27.81	24.00	58.0	91.5	14.3	13.7	+0.020	-0.025	37.30
1.750	2.055	1.760	28.03	24.28	57.9	90.9	14.4	13.4	+0.030	-0.030	37.50
4.000	4.415	3.865	55.47	47.22	59.3	87.7	12.5	12.8	+0.120	-0.075	339.0
4.000*	4.480	3.925	--	12.54	59.5	89.4	12.4	--	+0.070	-0.090	94.5
4.000*	4.480	3.970	--	10.99	59.5	91.1	11.4	--	+0.080	-0.065	86.5
4.000	4.500	3.915	55.26	48.38	59.7	90.1	13.0	12.5	+0.145	-0.075	366.0

*Indicates Two Bars Were Originally One (Broke in Green State)

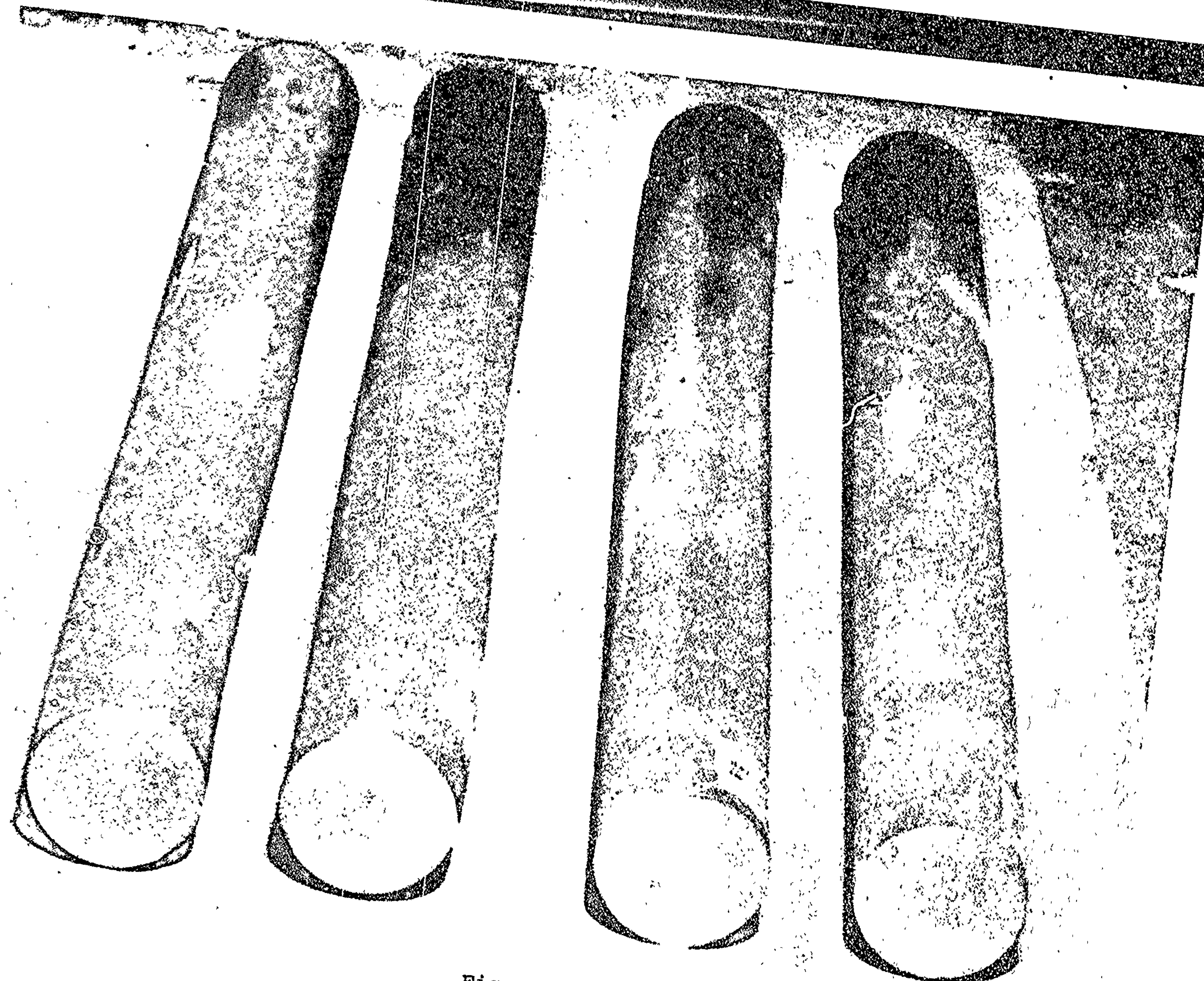


Figure 1

As-Received 1-3/4" Round Electrodes

were positioned away from the steel trunions to prevent melting of the structural components. The loaded rack was positioned in a dry box which was subsequently pumped down to approximately 5 microns. The box was then back filled with argon to slightly below atmospheric pressure. It was found that during welding, it was necessary to produce one continuous weld joint around the electrode. If, for any reason, the weld was interrupted, thermal cracks developed adjacent to the weld area, due to stress induced by shrinkage and contraction.

By using the above procedure, electrodes up to 2-1/2" in diameter were welded satisfactorily. Attempts were made to weld 4" diameter electrodes, but on each attempt thermal cracking initiated approximately 1" to 4" back from the weld area in both bars. Consideration was given to preheating the large diameter bars to permit satisfactory welding. However, since this would require special facilities to permit continuous heating during the welding operation, it was decided that mechanical joining be investigated prior to designing a new welding jig with auxiliary heating equipment.

Power requirements for the various size electrodes investigated are given in the following table:

TABLE IV

ELECTRODE WELDING POWER REQUIREMENTS

Electrode Diameter	Voltage	Amperage
1.00" - 1.5"	12-14	700-750
1.50" - 1.75"	12-14	750-800
1.75" - 2.00"	12-14	800-850
2.00" - 2.25"	12-14	850-900
2.25" - 2.50"	12-14	900-950

Figure 2 shows a typical as-welded electrode. The entire weld area was free of oxide stain indicating a good welding atmosphere. Figure 3 shows an intentionally broken electrode weld joint. The degree of bar shrinkage can readily be seen due to the welding operation. Also shown is the very limited depth of weld penetration.

b. Mechanical Joining

There are several methods of mechanical joining which could have been considered. However, since joining by threaded nipples on large diameter molybdenum electrode stock had been routine, this joining method was chosen for investigation.

Initial joining attempts involved drilling a hole to the size required for tapping, which, in the case of 4" diameter electrodes, had been established as 1.75" (7NC). However, severe tool chatter resulted and the vibration caused the electrode end to break. The next attempt involved drilling a small pilot hole and increasing the diameter in small successive increments. A 3/4" diameter pilot hole was accomplished satisfactorily, but severe tool chatter and tool wear were experienced. In attempting to increase this hole to 1" diameter by drilling, the electrode end cracked. It was concluded that the severe tool chatter was causing vibration and resultant cracking within the bar.

The next attempt was made by heating the bar to a temperature above the ductile-to-brittle transitions so as to not be as sensitive to tool vibration. Arbitrarily, the temperature requirement was set at 800°F. To determine the feasibility of this operation, an acetylene torch which was mounted on the lathe was used to heat the bar. A contact pyrometer was used to determine the temperature. When the bar reached 800°F, a 3/4" diameter pilot hole was drilled with significantly improved tool wear and machining

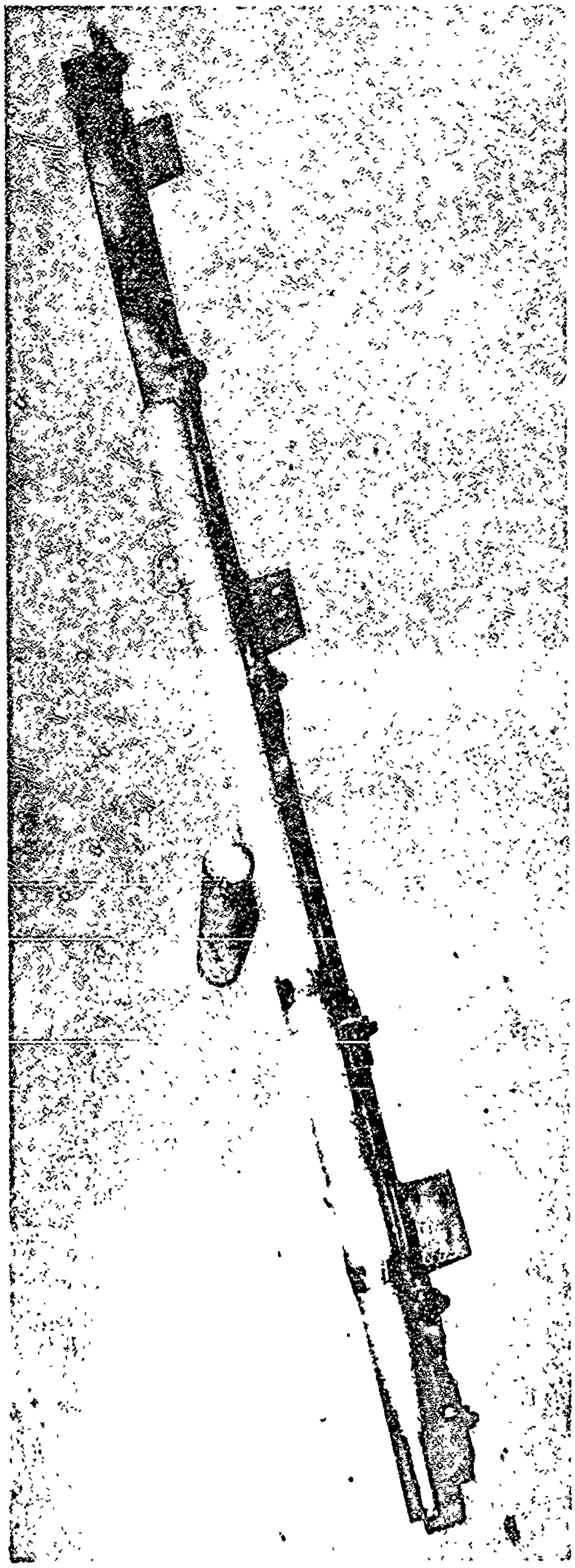


Figure 2

Welded 1-3/4" Diameter Electrode

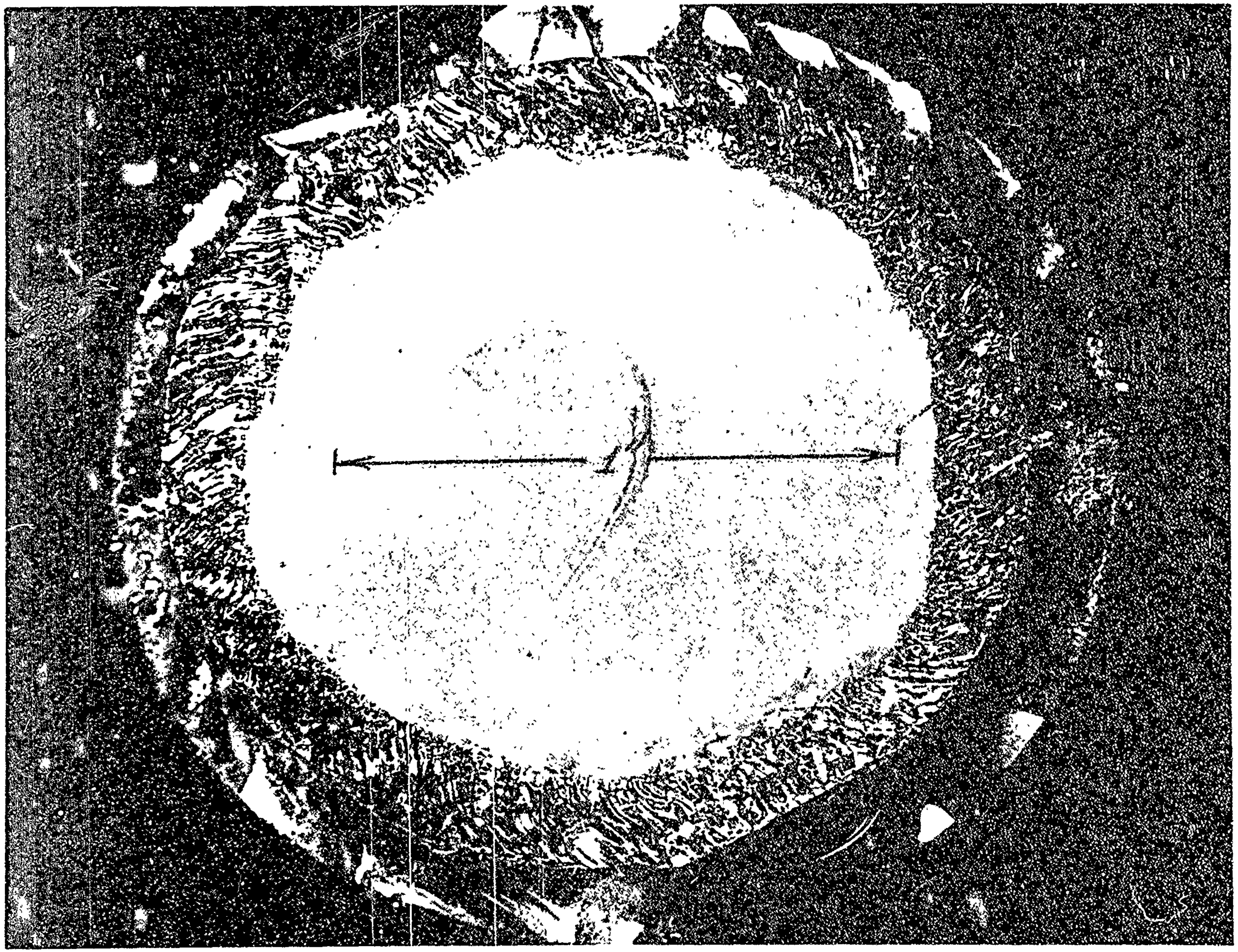


Figure 3

Electrode Weld Penetration

time, and tocl chatter was essentially eliminated. The hole was increased in size in 1/8" increments up to the final size for threading. It was noted that each time the bar cooled to approximately 700°F, chatter and vibration initiated. Attempts to thread the hole by using a standard 1.75" 7 NC tap proved unsuccessful. Satisfactory threads were accomplished only by again heating the bar to 800°F and machine threading. In this operation, temperature control was more critical as the threads chipped out when the temperature dropped below approximately 750°F.

Nipples for joining the large electrodes were machined from wrought powder metallurgy produced bar stock utilizing procedures similar to that for internal threading of the electrode, i.e. preheating and machine threading. No lubricant was used during any of the machining operations. Typical machined 4" diameter electrodes and nipples are shown in Figure 4.

B. Initial Vacuum Arc-Melting Studies

1. Melting Parameters

Experience previously established by Universal-Cyclops in melting small tungsten ingots, up to 5" diameter by approximately 12" long, was used as base line data for initial melting studies. The major problem for consideration in the initial melting studies was the attainment of a good sidewall to minimize yield losses in conditioning. Once an initial melting procedure has been established, it is usually possible to improve sidewall conditions by refinements in power input and adjustment of electrode to mold ratio. Since previous melting experience had indicated that the power settings were optimum for the particular electrode to mold ratio used (.4:1), a thorough investigation of electrode to mold ratio seemed the most promising for improving yield. Initial investigations, therefore, involved melting of 1.5", 1.75",

Figure 4

Machined 4" Diameter Electrodes and Nipples

2.00", 2.25", and 2.50" diameter electrodes into a constant mold size of 3.875" diameter.

2. Ingot Evaluation

One ingot was melted from each electrode diameter in order to determine melting characteristics, sidewall condition, and overall yield. Melting conditions for the various electrode diameters are given in Table V. The melting characteristics for each heat are given as follows:

a. Heat KC974

The 1.5" diameter electrode melted into the 3.875" diameter mold was the ratio most commonly used in previous melting experience by Universal-Cyclops. The melting characteristics of this heat were very good with a uniform melt rate and rather constant pressure. The last four minutes represented the hot topping cycle in which the power was uniformly reduced to 120 KW. During cooling, the ingot shrinkage in the mold was .125". Thus in the 3.875" tapered mold, the final ingot diameter was 3.700" at the top and 3.575" at the bottom, for an average of 3.638". The ingot was conditioned to a uniform diameter, defect-free surface at 3.250" with resulting sidewall yield loss of 20.2%. The hot topping cycle was sufficient to eliminate any shrinkage cavity thus minimizing cropping losses. The overall yield from ingot to conditioned billet was 61.3%. Contact and emersion ultrasonic inspection indicated the billet to be completely sound.

b. Heat KC978

Melting characteristics utilizing the 1.75" diameter electrode resulted in a very good pool formation during the entire melt even though the melting rate was somewhat slower than anticipated. The power utilized was also lower than anticipated and it was thought that a higher power level with resulting increased

TABLE V

MELTING CONDITIONS FOR VARIOUS ELECTRODE DIAMETERS

Heat No.	Electrode	Starting Pad
KC974	1.5" Ø x 49.63" long x 59.6 lbs.	3.75" Ø x 1.125" long x 8 lbs.
KC978	1.75" Ø x 50.56" long x 59.1 lbs.	3.75" Ø x 1" long x 7.5 lbs.
KC981	1.905" Ø x 37.63" long x 58.5 lbs.	3.75" Ø x 0.875" long x 6.5 lbs.
KC975	2.25" Ø x 22.41" long x 59.6 lbs.	3.75" Ø x 1.35" long x 10 lbs.

Heat No.	Pre-Melt Leak Rate	Pre-Melt Pressure	Pre-Melt Pressure Variation	Melting Time	Melting Rate	Power Required (Average)	Ingot Weight (lbs)	Conditioned Ingot Weight (lbs)
KC974	3 /min	5	5-12	25 min	2.35 lbs/min	209 KW	66.75	36
KC978	3 /min	2	3-15	32 min	1.75 lbs/min	200 KW	61.5	38.5
KC981	2 /min	4	5-12	37 min	1.59 lbs/min	210 KW	65.5	35.0
KC975	4 /min	4	5-8	27 min	1.55 lbs/min	220 KW	51.75	18.25

melting rate would have provided an even better ingot than that produced. The ingot sidewall was excellent except for one major defect at approximately mid-height. This defect correlated with the melting of the electrode joint, which in this heat was necessarily off-center due to a sharp camber in one of the electrode bars. Except for this one defect the ingot sidewall was satisfactory at 3.365" diameter for a resulting yield loss of 16.8%. The as-cast ingot with the defect indicated is shown in Figure 5. The conditioned billet yield was 71.3%.

c. Heat KC981

Melting characteristics of the nominal 2.00" diameter electrode and mold was too close to permit optimum melting conditions. The arc was erratic at times and the resulting sidewall was porous. The diameter of the conditioned defect-free surface was 3.140". The resulting sidewall yield loss was 25.5%. The conditioned billet yield was 59.3%.

d. Heat KC975

Melting characteristics of the 2.25" diameter electrode also indicated that the clearance between the electrode and the mold wall was too close for optimum melting conditions. Arcing to the mold wall was severe and two molds were burned through before successful melt was accomplished. Optimum power could not be reached because of the stray arc tendency and the resulting melting rate was extremely slow. Also, because of the slow melting rate and low power, a sufficient pool could not be maintained and the ingot sidewall was very poor. The ingot was conditioned to a defect-free surface at 2.906" with a resulting sidewall yield loss of 35.2%.

The following table gives the summary of billet yield resulting from each electrode size used:

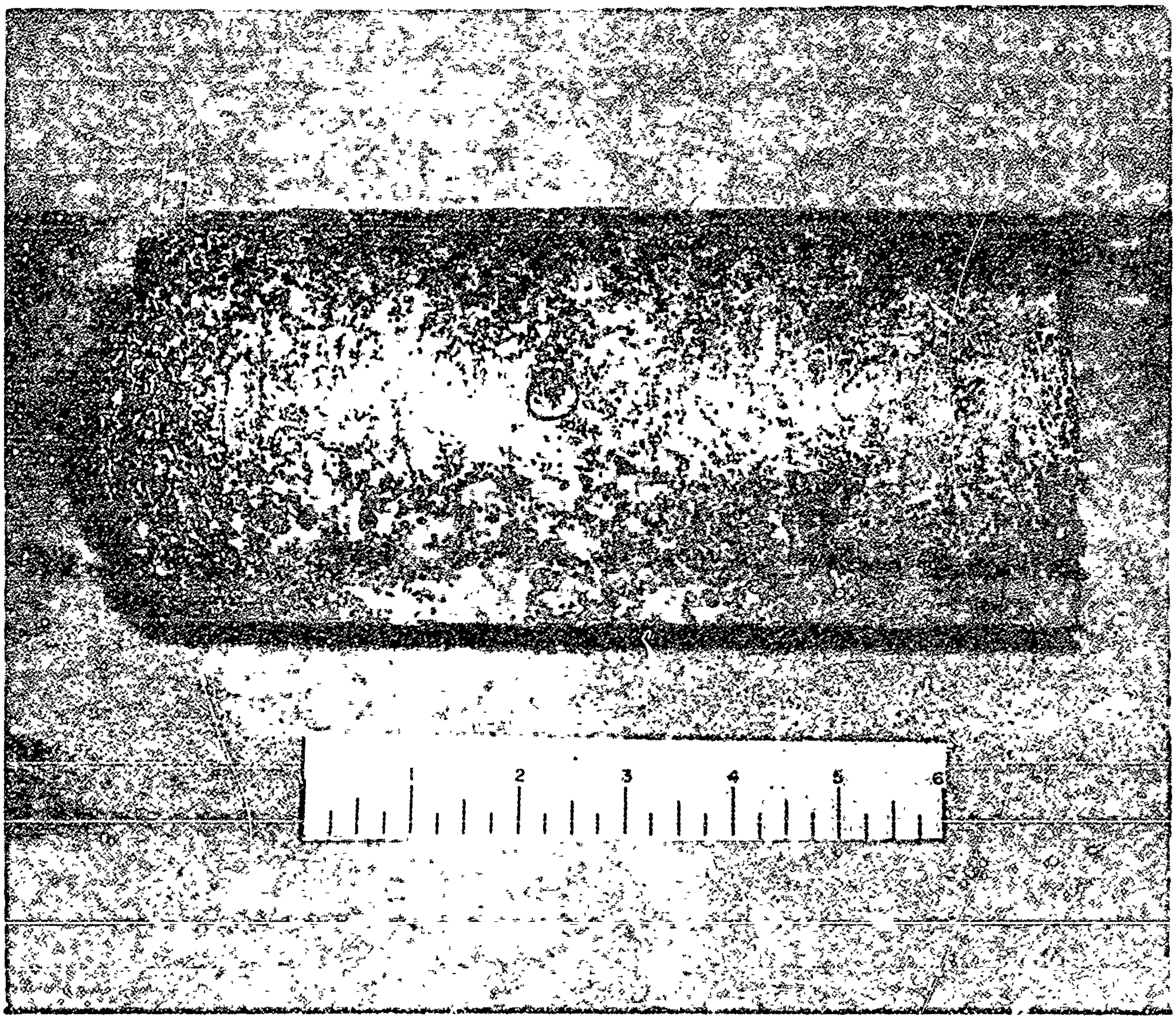


Figure 5

As-Cast Ingot, KC978
1.75" Diameter Electrode

TABLE VI

ELECTRODE SIZE VERSUS BILLET YIELD

Electrode Size	Billet Yield
1.50"	59.6%
1.75"	71.3%
2.00"	59.3%
2.25"	43.75%
2.50"	Not Melted

Due to the poor melting characteristics of the 2.25" diameter electrode, the 2.50" diameter electrode was not melted.

From evaluation of the above ingots, it was determined that the 1-3/4" diameter electrode represented the optimum size for melting into a constant mold size of 3.875" diameter based on yield results.

From these results, eight additional heats were melted using this electrode to mold ratio. All ingots had excellent melting characteristics and resulting sidewall condition. All melting parameters were very similar to Heat KC978 in which the 1-3/4" diameter electrode electrode was used.

The chemical analysis of the four experimental heats and six of the eight subsequent 3.875" diameter heats are listed in Table VII. From the table it can be seen that although one powder lot was used for all heats, there is a considerable variation in carbon and oxygen analysis. Since the majority of these heats were melted under identical conditions, it appears that inhomogenious powder and/or analytical techniques are responsible. Analytical technique is probably the principle factor since gas analysis techniques have not been refined to the point of reliability.

TABLE VII

ARC-CAST TUNGSTEN CHEMICAL ANALYSIS
(Results in ppm)

Ingot	Diameter	C	O	N	H	Fe	Si	Mo	As	Mn	Sn	Pb	Al	Cr	Ni	Co	Cu	Mg	Na	K	Ca
KC974	3.875"	45	4	8	2.3	8	20	100	100	10	20	<20	10	10	1	5	1	1	1	<1	1
KC975	3.875"	45	5	10	3.4	6	20	25	100	10	20	20	10	10	1	5	1	1	1	<1	2
KC978	3.875"	48	3	12	2.4	4	20	180	100	10	20	20	<10	10	1	5	1	1	<1	1	1
KC981	3.875"	42	3	8	1.2	2	<20	45	<100	10	<20	20	<10	10	1	<5	1	<1	1	<1	1
KC997	3.875"	21	21	15	<1	6	20	60	100	10	<20	20	10	10	1	5	1	1	<1	<1	1
KC998	3.875"	18	29	10	1.2	6	20	60	100	10	<20	20	10	10	1	5	1	<1	<1	<1	<1
KC999	3.875"	16	23	20	<1	10	20	20	<100	<10	<20	<20	10	<10	1	<5	1	<1	<1	<1	<1
KC1000	3.875"	13	16	16	<1	10	20	20	100	<10	<20	20	10	10	1	<5	1	<1	1	<1	<1
KC1002	3.875"	21	11	14	<1	6	<20	60	<100	<10	<20	<20	10	10	1	5	<1	<1	1	<1	<1
KC1003	3.875"	15	5	12	<1	3	20	20	<100	<10	20	<20	<10	10	1	5	1	<1	<1	1	<1
KC1135	5"	35	5	38	1	1	20	35	<100	<10	20	<20	<10	10	1	5	1	1	1	<1	1
KC1151	5"	36	6	25	2.1	11	20	36	100	<10	<20	20	10	<10	<1	5	1	1	<1	1	1
KC1158	5"	40	9	8	2.2	6	20	40	100	<10	<20	20	<10	<10	1	5	1	1	1	1	1
KC1160	5"	40	13	15	2.1	8	20	40	100	<10	<20	20	10	10	1	5	1	1	1	<1	1
KC1161	5"	36	17	32	5	8	20	17	100	<10	<20	<20	<10	10	1	5	1	1	<1	<1	<1
KC1175	5"	17	20	23	<1	3	20	46	<100	<10	<20	<20	<10	<10	<1	<5	1	<1	1	1	<1
KC1176	5"	10	20	25	<1	4	<20	40	<100	<10	<20	<20	<10	<10	1	<5	1	<1	<1	<1	<1
KC1178	5"	15	6	19	<1	5	<20	56	100	10	20	20	10	10	<1	5	<1	<1	<1	<1	<1
Average		28.5	12	17.2	1.65	6.1	<20	59	<100	<10	<20	<20	<10	10	<1	<5	<1	<1	1	<1	<1
Electrode Analysis		55	7	10	19	19	<20	85	<100	<10	<20	20	<10	<10	8	38	50	20	10	13	3

3. Destructive Ingot Evaluation

One ingot, Universal-Cyclops Heat No. KC1001, was ..ed for destructive evaluation according to the outline in ... Samples were provided for the following investigations:

Chemical Analysis

Hot Top
Mid-Height
Bottom

Macro Discs

Top Transverse
Center Transverse
Longitudinal

Micro Samples

Edge, Mid-Radius, Center As-Cast
Edge, Mid-Radius, Center Heat Treated 2 Hrs at 2600°F
Edge, Mid-Radius, Center Heat Treated 2 Hrs at 2850°F
Edge, Mid-Radius, Center Heat Treated 2 Hrs at 3100°F

..dness Samples

Top Macro Disc
Center Macro Disc
.dge, Mid-Radius, Center Heat Treated 2 Hrs at 2600°F
.dge, Mid-Radius, Center Heat Treated 2 Hrs at 2850°F
.dge, Mid-Radius, Center Heat Treated 2 Hrs at 3100°F

Chemical analysis from the top, middle, and bot- ... are listed in Table VIII. Again, the analytical ... close evaluation of the chemical homogeneity. It ...wever, that both carbon and molybdenum are signiif- ... hot top.

...re 7 shows transverse macro discs from the ... areas. Note that the top disc contains ...rain structure than the center disc. This

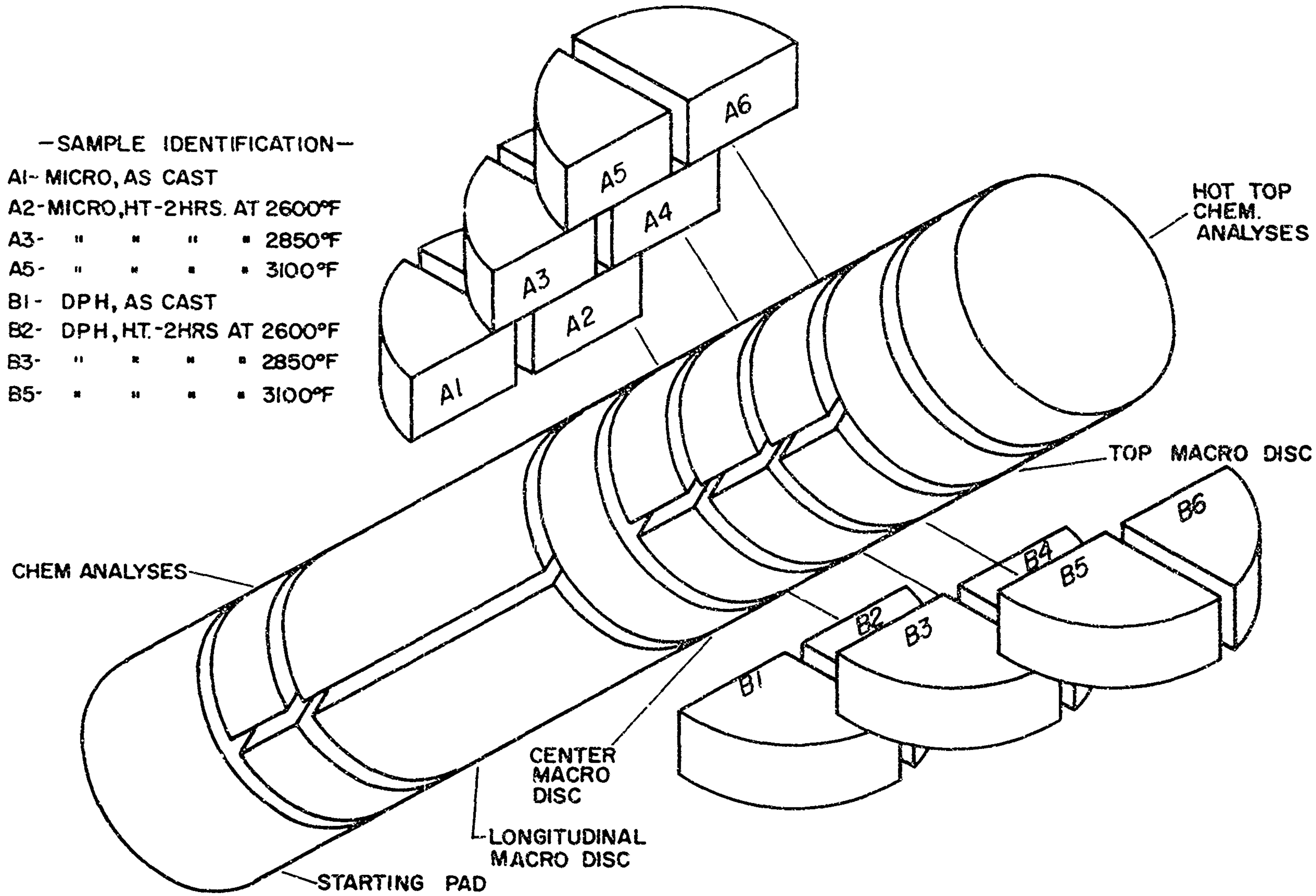


FIGURE 6
OUTLINE OF DESTRUCTIVE INGOT EVALUATION-KC 1001

TABLE VIII

CHEMISTRY ANALYSIS - HEAT KC1001

Location	C	O_2	N_2	H_2	Fe	Si	Mo	As	Mn	Sn
	Element									
Top	14	8	4	<1	3	<20	40	<100	<10	<20
Mid-Height	10	8	4	<1	6	<20	20	<100	<10	<20
Bottom	10	10	<3	1.2	3	<20	20	<100	<10	<20

	Pb	Al	Cr	Ni	Co	Cu	Mg	Na	K	Ca
Top	<20	<10	<10	<1	<5	<1	<1	6	<1	<1
Mid-Height	<20	<10	<10	<1	<5	<1	<1	6	<1	<1
Bottom	<20	<10	<10	3	<5	<1	<1	6	<1	<1

All Results in Parts Per Million

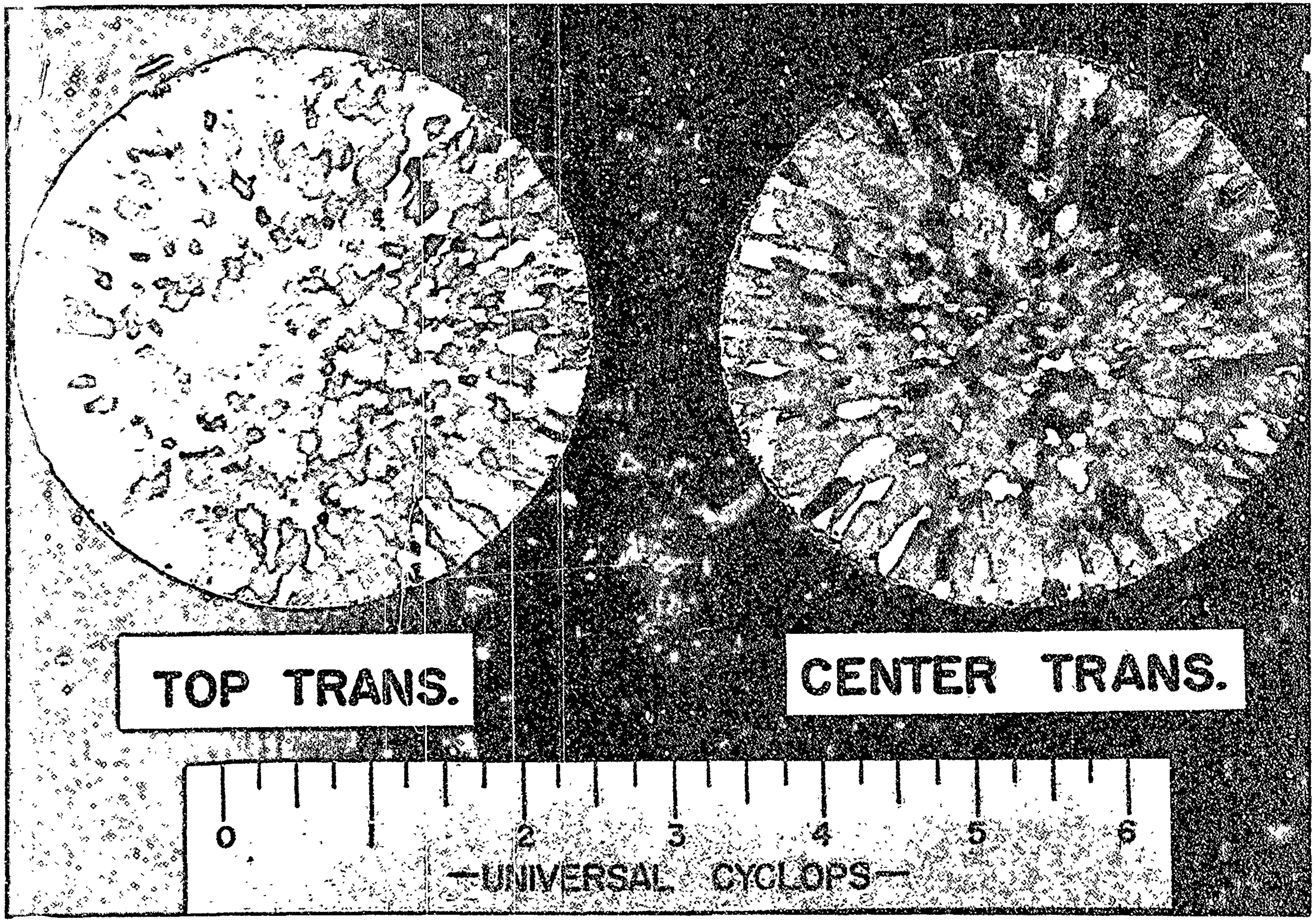


Figure 7

Macrographs of Transverse As-Cast Ingot Section - KC1001

can be attributed to the fact that the top section is freezing faster during the hot topping cycle due to the lower power input. The mid-height section represents the typical structure that is found throughout the remainder of the ingot.

Figure 8 shows a typical longitudinal structure. The void areas on the right hand mid-radius are grain voids pulled out during the cutting and polishing operation, and should not be construed as internal ingot defects. The macro slices in Figures 7 and 8 show that the degree of machining required to remove surface defects was very small.

The ingot hardness was measured on the center transverse and top transverse macro discs. The longitudinal hardness was measured on as-cast as well as the heat treated specimens. Table IX gives a summary of the ingot hardnesses under the various conditions investigated. Note from the table that a gradual decrease in hardness from edge to center is present in the center macro disc while the top disc is relatively uniform.

The as-cast longitudinal samples show that, although the surface is slightly harder than the ingot interior, the difference is not significant. Also, the mid-radius and center areas are very close in hardness which does not agree with the transverse readings.

The average hardness of the as-cast longitudinal samples is 353 DPH which correlates exactly with the lowest reading on the center transverse disc. In comparing these readings with the average on the top transverse disc, 371 DPH, it appears that the hot top is slightly harder than the other areas. This would tend to confirm the previous assumption that the hot top was a higher impurity area. Since heat treatments utilized did not result in any significant hardness change, it can be assumed that no noticeable degree of residual stresses are present in the as-cast structure.

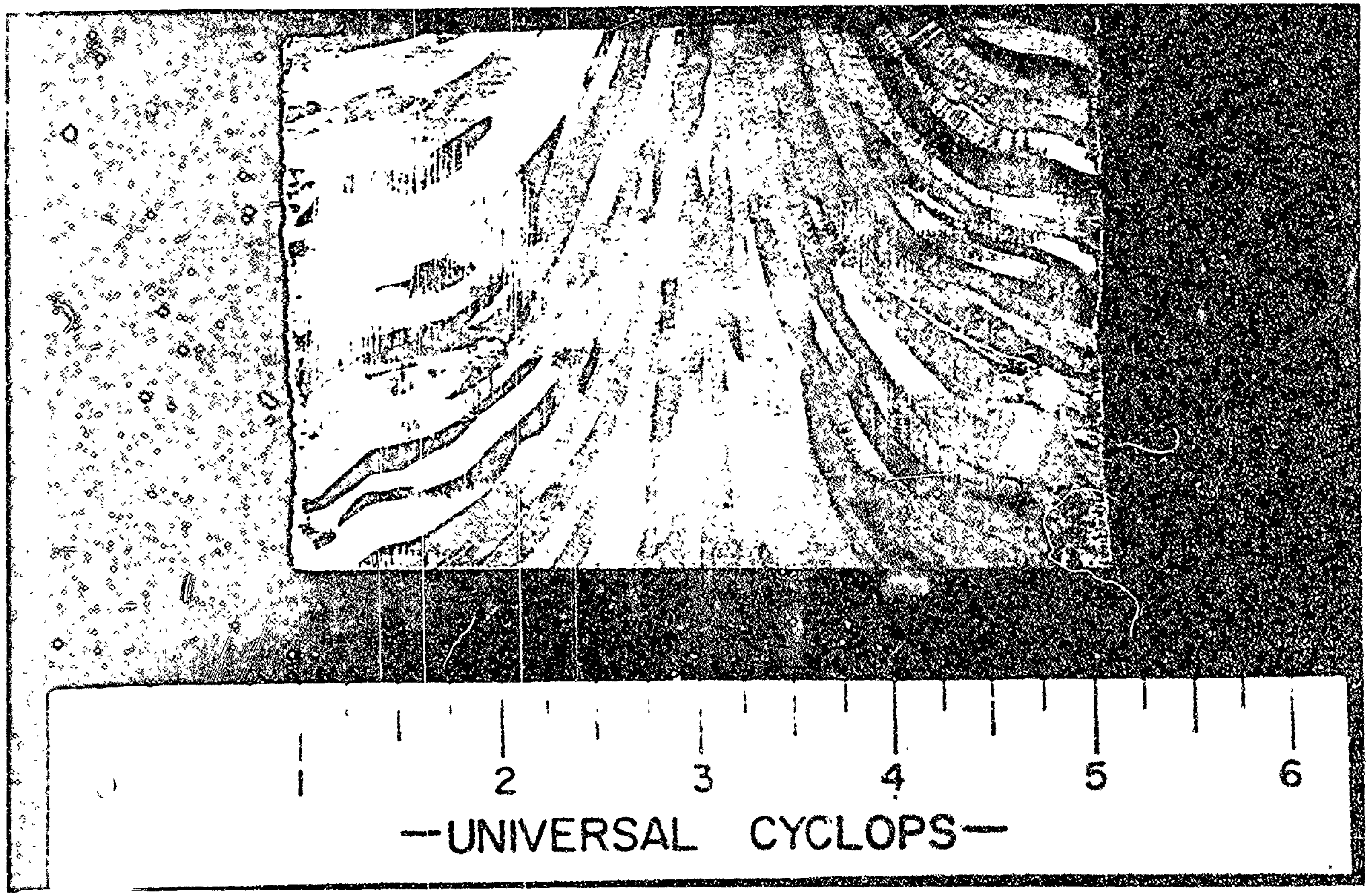


Figure 8

Macrograph of Longitudinal As-Cast Ingot Section

TABLE IX

INGOT HARDNESS SUMMARY
DIAMOND PYRAMID HARDNESS - 20KG LOAD

Sample Identification	E	EMR	MR	CMR	C	Minimum	Maximum	Average
Top Transverse	371	371	369	362	379	362	379	371.6
Center Transverse	384	379	374	368	353	353	384	370.4
Longitudinal As-Cast	361.5		347.3		350.5	347.3	361.5	353.1
Heat Treated 2 Hrs @ 2600°F	351.5		346		338	338	351.5	345.2
Heat Treated 2 Hrs @ 2850°F	341.6		353		360.6	341.6	360.6	351.7
Heat Treated 2 Hrs @ 3100°F	355		360		358.5	355	360	357.8

Code: E = Extreme Edge
EMR = One-Half Inch From Edge
MR = One Inch From Edge
CMR = One and One-Half Inches From Edge
C = Ingot Center

The two photomicrographs in Figure 9 represent the same area at low and high magnification. The first micro shows two primary grains, one with a clean matrix and the other containing a secondary grain boundary pattern. It was noted that in reviewing all specimens, a definite pattern of clean grains adjacent to those containing the secondary grain boundaries existed. The secondary grain boundaries are apparently dislocation sites visible because of the particular grain orientation shown. The second micro in this figure shows what appears to be two globules of a second phase. One has caused considerable deflection of the primary grain boundary during freezing.

Figure 10 depicts the ingot structure in the ingot center both as-cast and heat treated. The as-cast micro shows the secondary grain boundaries in the ingot center. The matrix within the sub-grain boundaries would appear to be a second phase but the purity of the material makes this questionable. The sample heat treated for two hours at 2600°F shows the secondary grain boundaries and matrix after heat treatment. Note that the particles are triangular in shape and are primarily unidirectional.

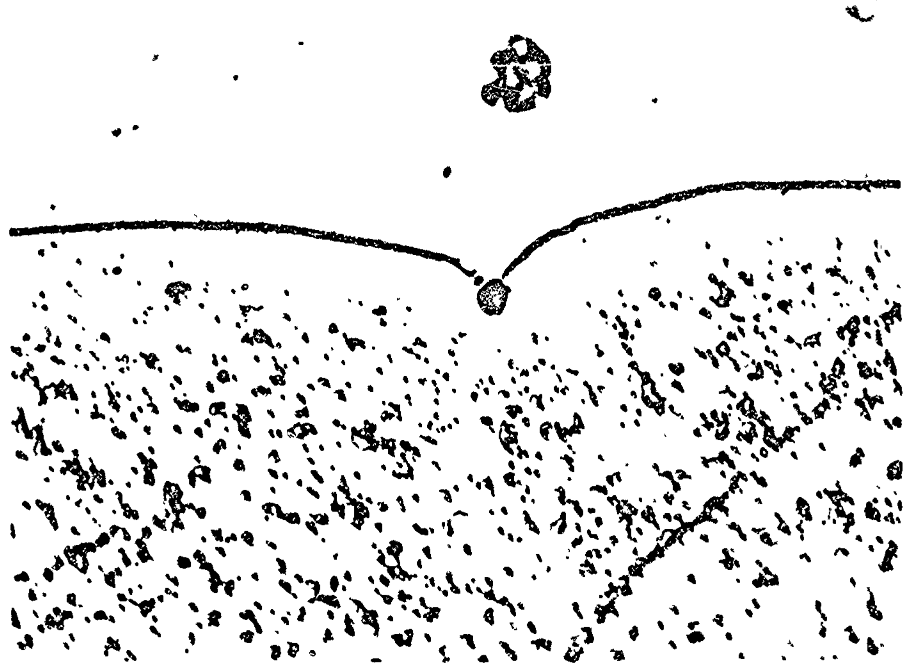
From the sample heat treated for two hours at 2850°F, it should be noted that the sub-structure above the primary grain boundary is divided into two areas separated by a long continuous secondary grain boundary. The matrix in these two grains is then separated into smaller sub-grains. The sample heat treated for two hours at 3100°F shows primary grain boundaries. Although this structure is different from that shown in previous micros, sub-grain boundaries were present in other areas of this specimen.

C. Scale-Up to 4" Diameter Conditioned Ingot Melts

In order to scale-up to the progressive phase requirements of the contract, it was necessary to develop melting techniques

As-Cast
Edge Location
Secondary
Grain
Boundaries

R9899 100X



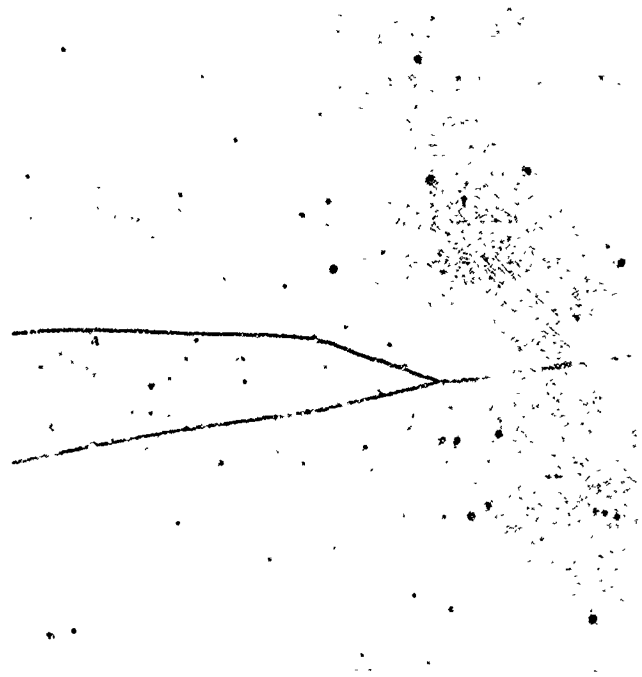
As-Cast
Edge Location
Possible
Second Phase
Grain Boundary
Impurity

R9898 1000X

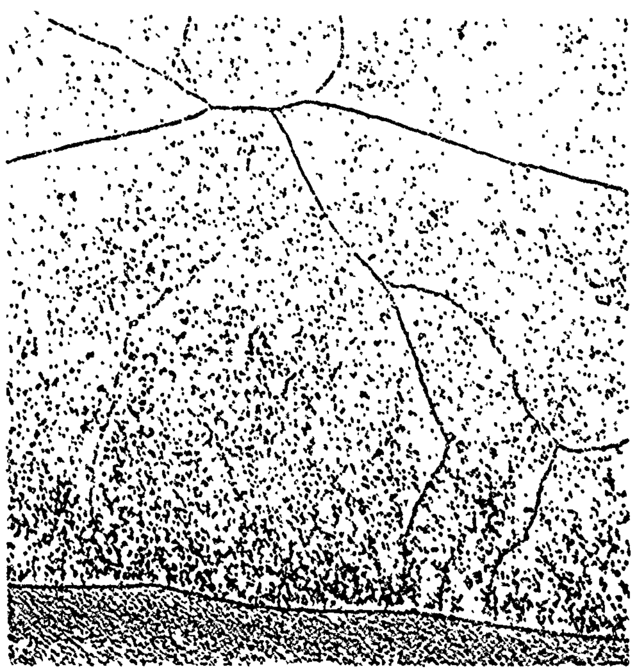
Figure 9

Arc-Cast Microstructure - As-Cast

R9903 200X
Arc-Cast Annealed 2 Hrs at
2850°F Mid-Radius Location



R9904 200X
Arc-Cast Annealed 2 Hrs at
3100°F Center Location



R9900 200X
Arc-Cast Center of Ingot

R9901 500X
Arc-Cast Annealed 2 Hrs at
2600°F Edge of Ingot

Figure 10

Arc-Cast Microstructure - As-Cast - Heat Treated

for a conditioned 4" diameter ingot to meet the 24" x 24" sheet requirements given by Phase III. Using electrode to mold ratios previously determined, a 2-1/4" diameter electrode was utilized for producing a 5" diameter as-cast ingot. The power required to melt these ingots was higher than anticipated and this, in conjunction with the problem of achieving only 80% of theoretically available power, resulted in poor melting conditions and extensive sidewall porosity on all of the six as-cast ingots produced. After conditioning to the 4" diameter extrusion billet size, five of the ingots were satisfactory with one being rejected for sidewall porosity. Chemical analysis for these five heats along with three additional heats used later on in the Phase III portion of the program is given in Table VII.

D. Scale-Up to 6" Diameter Conditioned Ingot Melts

The progressive phase requirements for the contract called for a scale-up to finish sheet size of 36" x 36" square. The minimum conditioned ingot size necessary to achieve a cross section compatible with this sheet size requirement was determined to be 6" diameter.

An initial attempt to melt the 4" diameter electrode shown in Figure 4 into an 8" mold resulted in a complete failure. During melting, 2" to 6" lengths of electrode continually dropped off into the pool. The melt was stopped after seven minutes with a resulting 300 pound scrap loss due to this breakage phenomena. The remaining electrode bar was ultrasonically checked for cracks. A crack was detected approximately 3" from the melting end indicating a progressive failure as the melting continued. The remainder of the bar was found to be crack-free. It was assumed that the rapid build-up of heat in the electrode due to resistance heating and radiation from the pool resulted in the cracking problem. In addition, melting furnace deficiencies caused erratic conditions and poor control.

Extensive modifications were made to the arc-melting furnace. These included additional power and modified power input, modified cooling and a change in the electrode feed mechanism. When these modifications were completed four ingots were melted into an 8" diameter mold. The melting conditions were essentially satisfactory; however, at intervals, the melt became erratic. This was attributed to the basic electrode as the conditions would initiate when proceeding from one bar to the next and would stop when this bar was consumed and the melting of the next bar initiated. A typical as-cast ingot is shown in Figure 11. The melting history and billet yields for these ingots are shown in Table X.

TABLE X

INGOT MELTING AND PROCESSING

	Heat 1147	Heat 1148	Heat 1167	Heat 1168
Mold Diameter (Inches)	8	8	8	8
Electrode Diameter (Inches)	3-1/4	3-1/2	3-7/8	3-7/8
Weight Melted (Pounds)	518	523	571	453
Conditioned Weight (Pounds)	231	233	301	153
Yield (Percent)	44.7	44.6	52.8	33.8

As indicated in the table, the yield values were relatively low. This is due largely to the fact that in order to insure a completely satisfactory ingot at the required 6" diameter, an 8" mold was used. In machining of these ingots, sidewall porosity was eliminated in every case at 7" to 7-1/4" so that a much higher yield could have been realized if a 7" extrusion container were available. Another area of appreciable yield loss was on the hot top of three of the four ingots. On three ingots, the average yield loss for hot top cropping only was 14.5%.

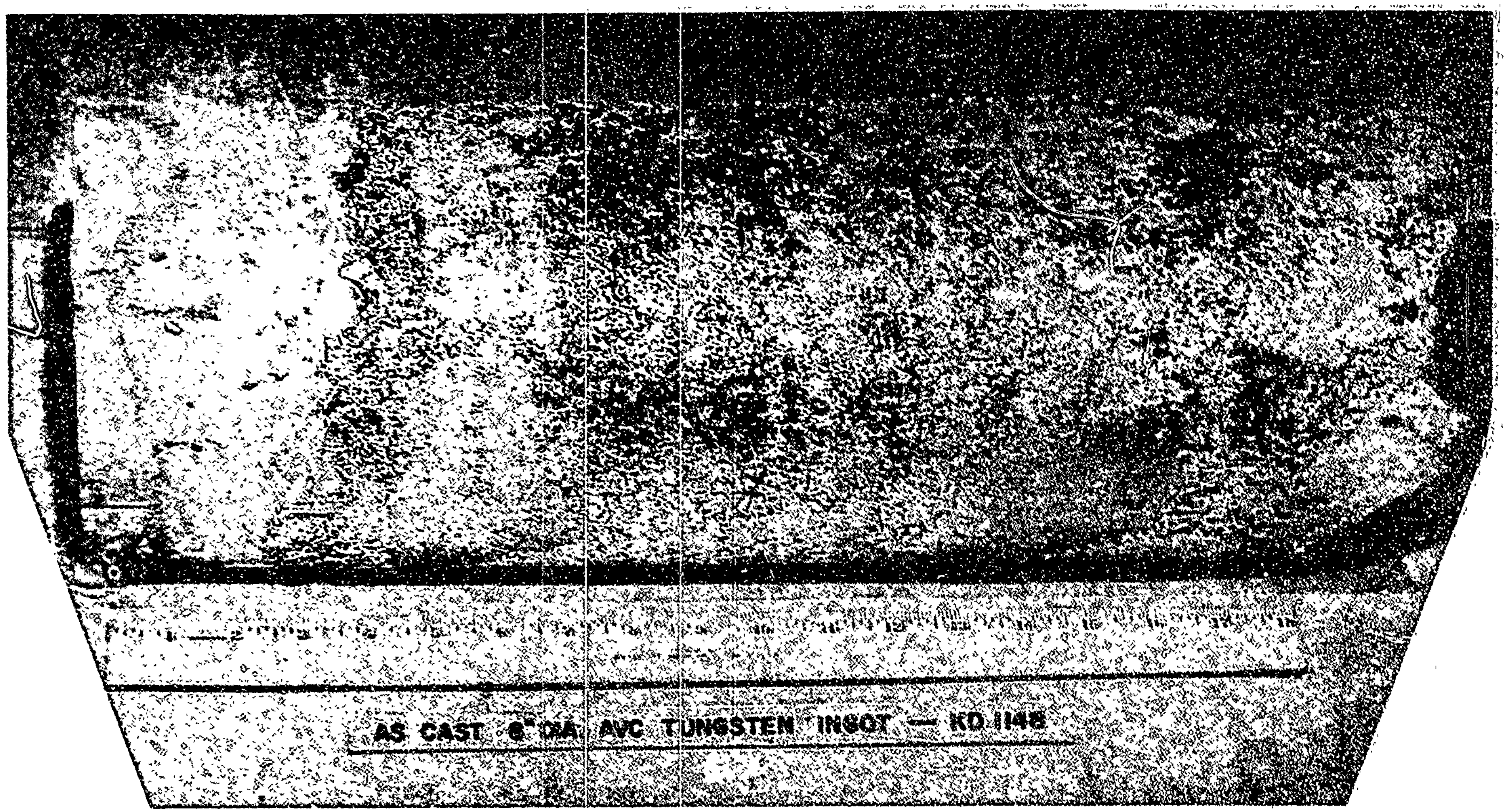


Figure 11

As-Cast 8" Diameter Ingot

Chemical analysis for the starting electrode and the ingot are listed in Table XI. In comparing the data, two elements merit discussion. In the second electrode lot, the nickel content is relatively high and well above the 20 ppm maximum specification level. Due to the time delay in rejection, the material was melted subject to rejection if the ingot chemistry was not satisfactory. As shown in the ingot chemistry for Heats KD1167 and KD1168, the nickel content, using these electrodes, was below the 1 ppm detection limit. Molybdenum content in the two powder lots is shown to be 8 and 11 ppm respectively. In the ingot chemistries, only one heat is below 100 ppm. This deviation between electrode and ingot chemistry has been a continuing problem and can only be ascribed to the inconsistencies in analytical techniques at these low impurity levels.

E. Scale-Up to 8" Diameter Conditioned Ingot Melts

In order to produce the final sheet requirements of this contract (36" x 96" at .020", .040", and .063") by the most economical method and to achieve the maximum flexibility in cross rolling, 8" diameter conditioned ingots were required. To acquire this 8" diameter conditioned ingot size, a 9-1/2" diameter mold was fabricated and electrodes were ordered for two heats, one to be melted with 4-1/2" diameter electrodes, and the other with 4-3/4" diameter electrodes. For each heat, three bars were required to produce the desired electrode weight and these were assembled by machining 1-3/4" 7 NC female threads in the bars and connecting them with male nipples. The 4-3/4" diameter bars were assembled for the first melt. Approximately half way through the first bar, the entire electrode assembly sheared off at the top joint and fell into the pool. Examination revealed that a crack initiated at the root of the female thread in the top bar. The problem appeared to stem from three possible sources, all based on the notch-sensitivity of the female threads:

TABLE XI

ELECTRODE AND INGOT CHEMICAL ANALYSIS

Electrodes

Lot	Element											
	As	Al	Ca	Cr	Cu	Fe	K	Mg	Mo	Na	Ni	Si
964	<3	0.6	6	2	0.1	4	<20	<2	8	7	6	3
996	<3	9	6	18	0.1	14	35	<2	11	20	45	3

Ingots

Ingot	Element															
	Mn	Al	V	Cr	Cu	Fe	Co	Mg	Mo	Ti	Ni	Si	C	O	N	H
KD1147*	<10	<10	<10	<10	<1	13	<5	<1	100	<1	<1	<20	30	14	20	1.9
KD1148*	<10	<10	<10	<10	<1	13	<5	<1	500	<1	<1	<20	30	11	21	1.6
KD1167**	<10	<10	<10	<10	<1	12	<5	<1	<10	<1	<1	<20	10	9	2	<1
KD1168**	<10	<10	<10	<10	<1	5	<5	<1	190	<1	<1	<20	44		14	

*Electrodes from Powder Lot 964

**Electrodes from Powder Lot 996

All Analyses in ppm (<) Indicates Analysis was Below Detection Limits

1. Increased stress on the thread due to overall increased weight of the 4-1/2" diameter electrodes.

2. Electrode vibration due to initial power surge on melting.

3. A lower relative density in the thread area due to larger cross section over the bars compared to those previously used in smaller heats.

The electrode design was changed to produce future electrodes with hollow centers in order to achieve a higher density in the thread area. Also, attempts to decrease the notch sensitivity were made by incorporating a slight radius on the thread root.

The 4-1/2" diameter solid electrodes were then melted without incident to provide a 985 pound as-cast ingot. This ingot was machined and ground to an approximate billet diameter of 7.840" with a finish weight of 640 pounds representing a 65% yield from as-cast to conditioned ingot. Ultrasonic examination indicated the billet was completely sound. Figure 12 shows the conditioned billet ready for extrusion.

Based on the above information, 4-1/2" OD x 7/8" ID electrodes were ordered in sufficient quantity to produce eleven 8" diameter conditioned billets at a nominal 1000 pound average per ingot. During the melting operations on the eleven production heats, it was noted that extreme changes were occurring in the melt rate during melting. On previous heats, a normal voltage fluctuation was established at approximately ±4 volts. During melting of the 4-1/2" diameter electrodes, the voltage fluctuation would abruptly change to a nominal ±1 with a corresponding melt rate increase of 50% to 100%. It was originally believed that these changes were occurring at the joints between the electrode bars. However, by plotting electrode travel on timed voltage tracts, it was shown

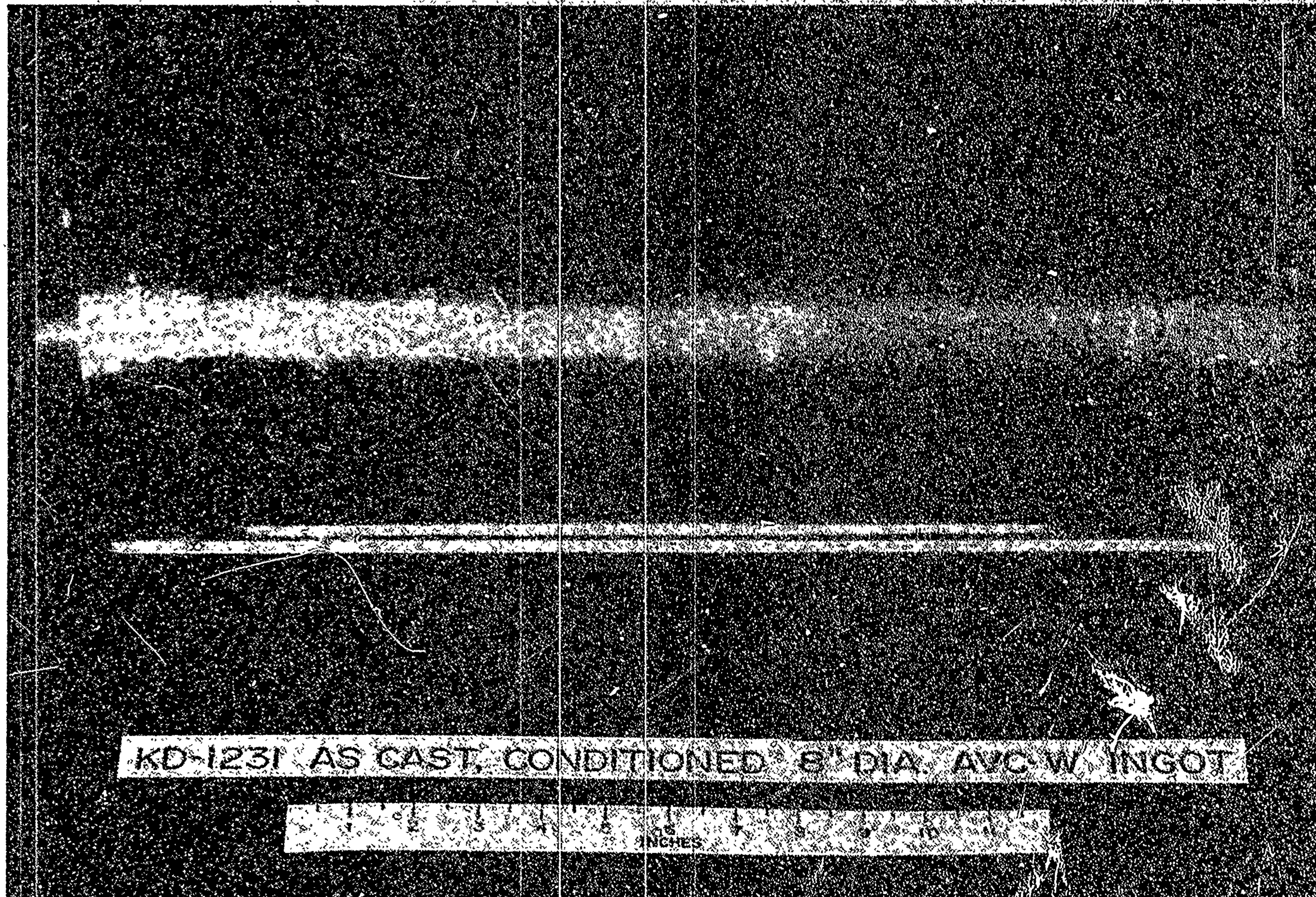


Figure 12

Conditioned 8" Diameter Billet

that the changes did not necessarily occur at a joint. The two principle factors which would cause this effect were electrode density and purity. However, no correlation could be developed from the available electrode information. This erratic melt behavior resulted in localized areas of porosity on the conditioned 8" diameter ingots generally correlating with the high melt rate areas.

The eleven production ingots were preheated to 1000°F and subsequently annealed at 2400°F followed by a slow cool in vermiculite. They were then machined to the 8" diameter extrusion size and ultrasonically inspected. The ultrasonic inspection revealed that two of the ingots were internally cracked throughout the length. In an attempt to determine when the ingot had cracked, they were fractured in half by cold forging. It was assumed that if the cracks occurred either during melting or annealing, it would be oxidized from the exposure to air after annealing and conversely if they were not oxidized, the cracking had occurred during machining or cutting. Visual examination of the fractured ingot revealed that discoloration was present, but the crack did not extend to the periphery, the mode of cracking apparently proceeding upward from the pad. It was concluded that either during ingot solidification or thermal expansion and contraction during annealing, crack propagation progressed from the pad through the ingot, with the latter being the most probable cause.

III. Ingot Breakdown Evaluation

The two most common methods for ingot breakdown are direct forging and extrusion. The state-of-the-art analysis indicated that no known attempts have been made at direct forging of as-cast unalloyed tungsten ingots. It was considered, however, that the high purity of the arc-cast material and the absence of high carbon grain boundaries would permit direct forging if the optimum conditions could be established. Considerable experience had been developed on extrusion of arc-cast tungsten and tungsten-base alloys. In order to best meet the final contract objectives, ingot breakdown investigations were made by 1) direct forging, 2) direct extrusion, and 3) InFab* forging, with the most promising method incorporated for a scale-up to final size requirements.

A. Direct Forging

As a result of the survey, one company reported considerable success in closed die forging of arc-cast tungsten into a typical flower pot shaped rocket exhaust nozzle. This organization was contacted and a program outline to investigate direct forging of ingots to sheet bar. Since the dimensional requirements for sheet bar was not compatible with closed die forging, the investigation was conducted on flat dies not restricting sidewall movement. The investigation was initiated to determine criticality of the elasticity range over a variation of temperatures and reductions.

1. Procedure

Three temperature levels were selected for investigation, 2500°, 2750°, and 3000°F. The schedule called for an initial upset of 20%, evaluation of soundness, and subsequent

*An inert atmosphere fabrication facility built by Universal-Cyclops under Contract NOa 55-006-c designed to forge and roll refractory metals at temperatures up to 4000°F.

reforging. Heating was accomplished using a gas fired furnace. Utilization of preheated air (1700°F) for the air-gas mixture, permitted rapid increase in furnace temperature and extremely high temperature furnace capabilities up to 3500°F.

2. Forging Parameters

A total of five ingot sections were selected for forging evaluation. The forging parameters are shown in Table XII.

TABLE XII

FORGING PARAMETERS

	Forging A	Forging B	Forging C	Forging D	Forging E
Material	KC981*	KC981**	KC978**	KC978*	KC975
Furnace Temperature	2500°F	2750°F	3000°F	2750°F	2750°F
Preheat	700°F	700°F	700°F	700°F	700°F
Transfer Time	15 sec	17 sec	14 sec	16 sec	15 sec
Finish Temperature	1950°F	2250°F	2425°F	2275°F	2450°F

*Bottom Half

**Top Half

3. Forging Evaluation

The five forged and sand blasted billets are shown in Figure 13. Also depicted are macrographs after being sectioned, polished, and macro etched.

a. Forging A

Four relatively light blows were used to upset the billet an estimated 20%. The billet was rotated 20° to 30° between each blow to prevent side shearing. The actual measured reduction was 20.5%. The forging was cleaned and slight side cracks

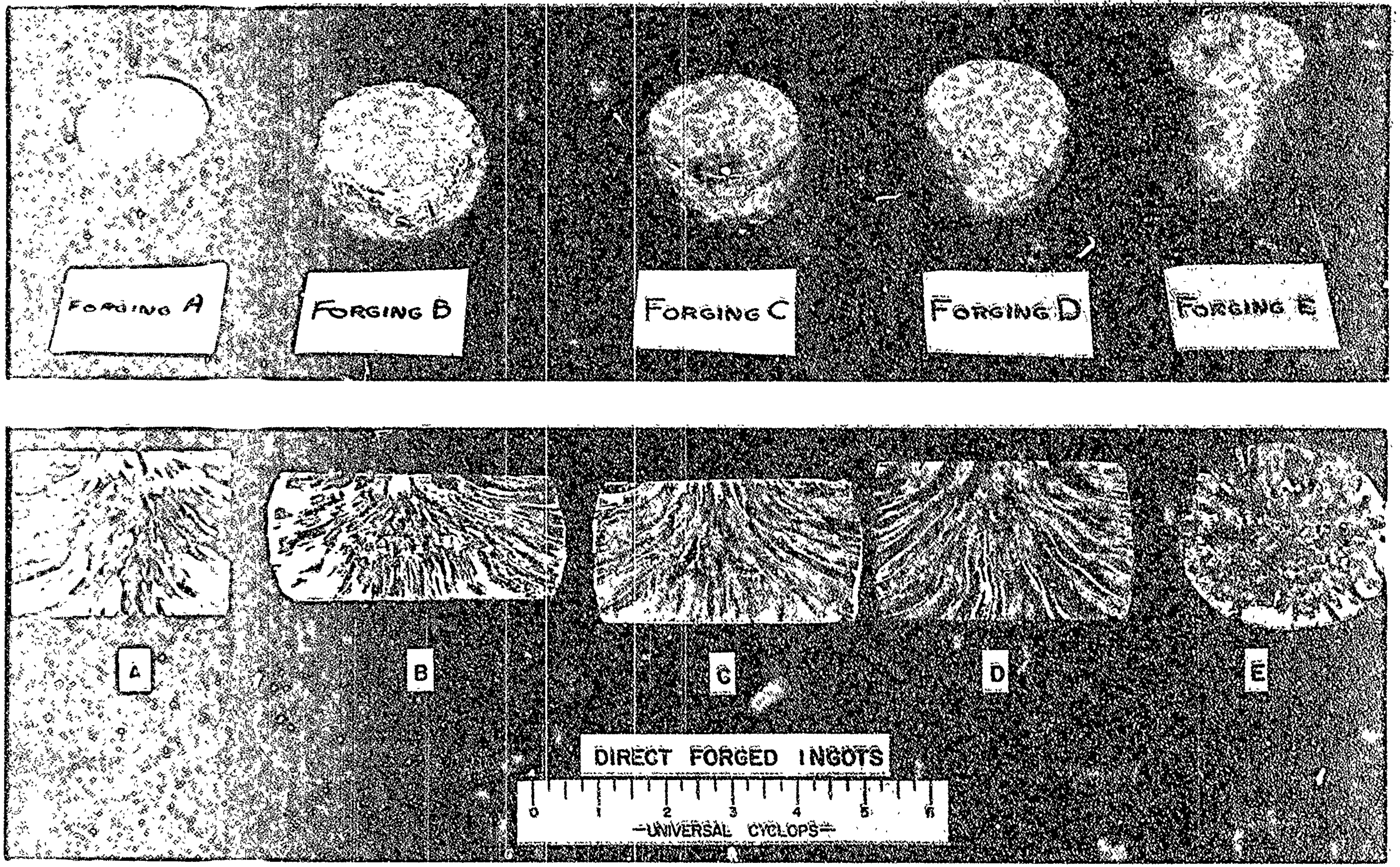


Figure 13

rect Forged Ingots - As-Forged and Longitudinally Sectioned

were observed. It was machined to a defect-free surface and again heated for additional forging. On the first blow of the second forging operation, the billet cracked severely at several locations along vertical planes of the periphery.

b. Forging B

Again four blows were given with rotation of the ingot between each blow. The billet was sound after approximately 20% reduction and was reheated to the original temperature of 2750°F. Forging was again initiated and an additional 20% reduction taken. Examination revealed cracks on the periphery running parallel to the direction of forging. Measured reduction by upsetting was 42.8%.

c. Forging C

This billet was forged using the same parameters, except temperature, as the previous billets. No cracking occurred during the initial 20% reduction. However, cracking did occur on the second forging operation after reheating to 3000°F. Total reduction at the point of visible crack initiation was 30.5%.

d. Forging D

The reduction of Forging B at 2750°F showed the best results of the three forging temperatures investigated. It was indicated that a 35% reduction could be taken safely after which a heat treatment would be required. This proved inaccurate when Forging D cracked after 22.5% reduction after being forged under identical conditions to Forging B.

e. Forging E

On this billet, radial forging was attempted, using "V" dies. After two blows, severe cracking was observed on the circumference as well as the ends. No measured reduction was obtained on this billet.

The forgings were further evaluated by micro and hardness determinations. High temperature heat and subsequent hardness measurements were also determined to study recrystallization phenomena. Heat treating ... in vacuum at 3000°, 3200°, 3400°, and 3600°F for ... Table XIII shows the pre- and post-hardness for each ... This investigation was limited to Forgings B and C which ... the most reduction. Note that in Forging B which ... reduction, the average as-forged hardness is 450 compared to the average as-cast ingot hardness of 372. Upon ... samples from this forging, the hardness dropped to a low ... DPH at 3400°F. Macro observation in this specimen ... recrystallization initiated during the lowest temperature ... successive anneals resulted in added recrystallization. ... which were slowest to recrystallize as shown by hardness examination were at the center of the forging near the ... surfaces.

Forging C had received only 30.5% reduction and ... showed that the as-forged hardness was significantly ... Forging B. The average as-forged hardness was 64 DPH ... the as-cast ingot. The resulting drop in hardness ... treatment was more gradual than on Forging B. Macro ... showed that recrystallization also initiated on this ... the lowest temperature anneal. Center areas near the ... surfaces did not completely recrystallize under any ... annealing conditions.

In studying the cracking problem on the forgings, ... observation clearly showed that the cracks occurred at the ... boundaries at all four forging temperatures. Micro examination ... showed that although grain boundary cracking had occurred, ... cleavage cracks were also present. The results of the forging

TABLE XIII

FORGING HARDNESS SUMMARY

AS-FORGED AND HEAT TREATED

Sample	As-Forged*	Heat Treated 3000°F	Heat Treated 3200°F	Heat Treated 3400°F	Heat Treated 3600°F	As-Cast Comparison
Forging B						
Edge	500	369	349	349	394	384
Mid Radius	450	369	337	337	381	379
Center	427	399	365	349	353	353
Average	459	379	349	345	376	372
Forging C						
Edge	462	381	349	347	349	
Mid Radius	430	415	379	362	362	
Center	416	391	367	383	379	
Average	436	396	365	365	362	

*Average As-Forged Hardness of Four Samples From Each Forging

studies indicated that successful forging could be accomplished by utilizing a small initial reduction with subsequent recrystallization and then continued forging. However, this procedure was not deemed desirable for scale-up because of heating and cooling problems for the large size billets.

B. Extrusion

From the state-of-the-art analysis, it was evident that considerably more experience was available on the extrusion of arc-cast tungsten and tungsten-base alloys than on direct forging of these materials. From the background information, the extrusion technique appeared to offer the greatest potential towards meeting the objective of initial breakdown requirements.

1. Procedure

Six extrusions were produced for initial breakdown studies. A typical extrusion billet 3.060" diameter by 6" long was conditioned from a 4" diameter arc-cast ingot produced from the initial melting investigation under this program. All surfaces of the billet were ground to provide optimum conditions for the extrusion operation. The billets had a finish of approximately 20 RMS as compared to an as-machined billet surface of approximately 175 RMS. A 1/2" 45° taper on the nose was provided to prevent or minimize the possibility of stalling the extrusion press prior to breakthrough. All initial extrusion studies were accomplished at the TAPCO Division of TRW, Inc. The press utilized had been modified especially for refractory metal development and a brief description of the facility is given in Appendix III to this report.

2. Extrusion Parameters

As extrusion has already been proven as a successful method for initial breakdown, the objectives in this study were

1) correlation of extrusion temperature with press capacity, resultant surface condition, and billet yield, and 2) direct extrusion to sheet bar.

The first three billets were extruded to nominal 1.5" diameter rounds. It was originally planned to utilize three successively lower extrusion temperatures starting at 3000°F. This sequence had to be changed, however, because the pressure required for the second billet, extruded at 2800°F, indicated that lower temperature would cause the press to stall. The third billet was extruded at 2800°F but slower speed was used than that for the second billet.

The second three billets were extruded to sheet bar at successively lower temperatures starting at 3250°F. The extrusion temperature was raised due to the fact that the extrusion ratio for sheet bar was appreciably greater than that for the rounds (6.6:1 versus 4.5:1). The pressure required for the second billet was essentially the same as that for the first even though the temperature was 100°F lower. The third billet extruded 200°F lower than the first required appreciably less pressure. A summary of the extrusion parameters is listed in Table XIV.

3. Extrusion Evaluation

The three extruded rounds are shown in Figure 14. From visual observation no nose bursting occurred on any of the three extrusions. Macro examination of the cropped noses revealed that both 2800°F extrusions had a micro crack from the edge to the center extending approximately 3" back from the nose. The remainder of these two extrusions were sound. The first billet, which was extruded at 3000°F was completely sound. The as-extruded surface of all the extrusions was not completely satisfactory in that frequent surface tears occurred, especially on the trailing edge of

TABLE XIV

EXTRUSION DATA FOR 3.060" BILLETS

Billet Identification	A	B	C	D	E	F
Billet Weight	29 lbs. 13 oz.	32 lbs. 8 oz.	32 lbs.			
Extrusion Shape	1.5" dia.	1.5" dia.	1.5" dia.	2" x 0.6"	2" x 0.6"	2" x 0.6"
Reduction Ratio	4.5:1	4.5:1	4.5:1	6.6:1	6.6:1	6.6:1
Temperature (°F)	3000	2800	2800	3250	3150	3050
Pressure						
Breakthrough	124,000	145,000	135,000	160,000	160,000	150,000
Minimum	112,000	124,000	124,000	-	-	-
Speed (IPS)	11.8	10.3	9.1	6	7	8
Die Size	1.461" dia.	1.471" dia.	1.468" dia.	1.954" x .595"	1.997" x .582"	2.003" x .557"
Billet Size						
Lead	1.510" dia.	1.522" dia.	1.520" dia.	2.037" x .630"	2.041" x .625"	2.041" x .642"
Middle	1.555" dia.	1.528" dia.	1.593" dia.	2.067" x .659"	2.050" x .636"	2.059" x .675"
Tail	1.558" dia.	1.535" dia.	1.595" dia.	2.137" x .840"	2.188" x .822"	2.207" x .825"
Length	24"	26"	25"	30.5"	35.5"	36.5"
Yield	88.5%	84%	85%	83.7%	86%	85%

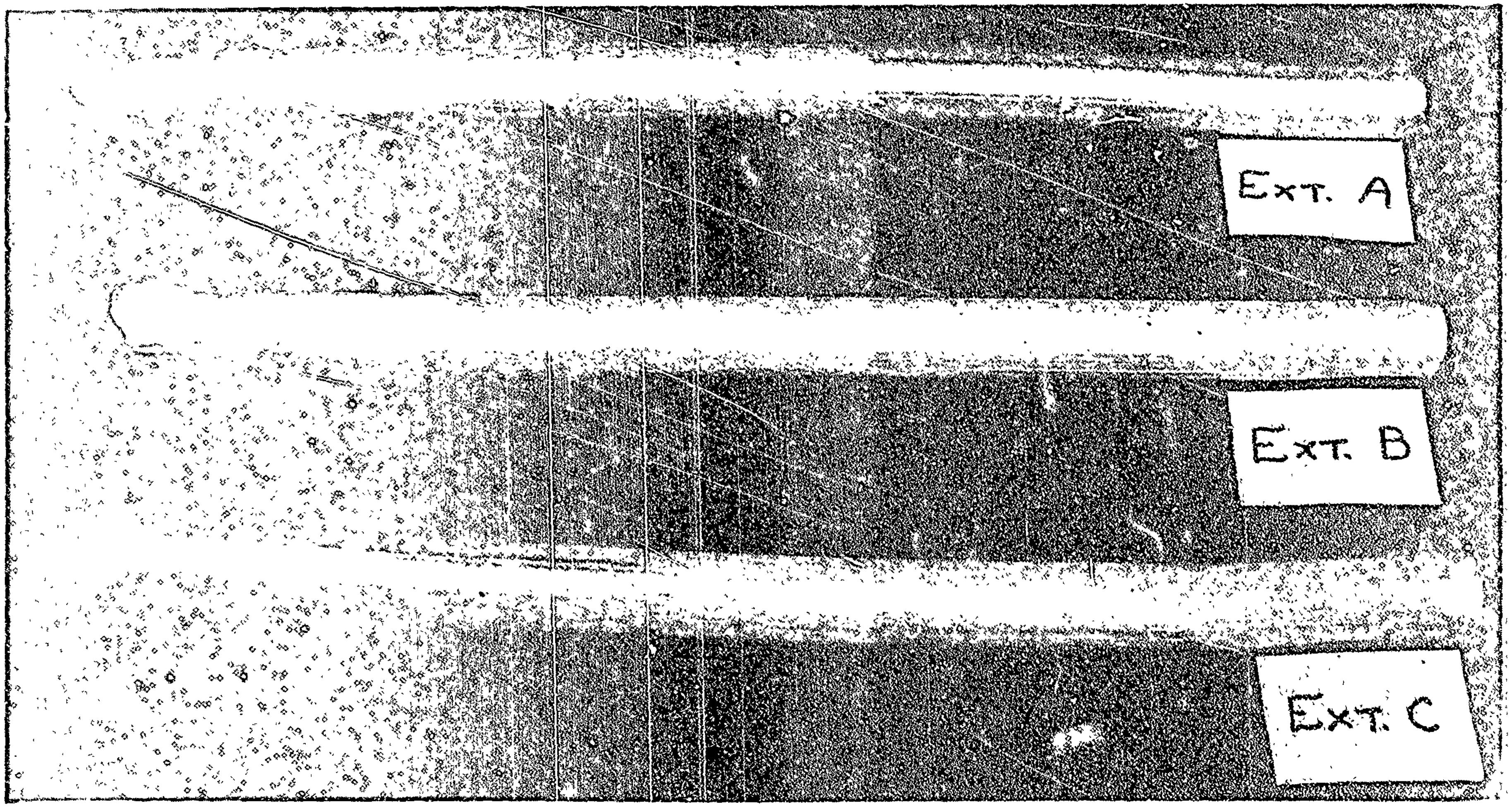


Figure 14

1.5" Diameter As-Extruded Rounds - From 3.060" Billet

the extrusions. No correlation between extrusion temperature and surface finish could be made.

The three sheet bar extrusions are shown in Figure 15. Again, it is noted that no visible nose bursts occurred. Micro examination also showed that the nose ends on all sheet bars were sound.

Figure 16 shows macro slices from the nose and tail ends of each of the six extrusions. The structures shown are typical of any highly worked transverse macro. Although the grain structure is not well defined, it will be noted that the sheet bars extruded at a higher reduction ratio do appear to have a finer grain size. The variation in the cross section of the sheet bar extrusions is due to severe die wash during the extrusion operation. This was a major disadvantage of the direct extrusion to sheet bar.

Samples were taken from nose and tail of each extrusion for heat treatment investigations. Evaluation consisted of metallographic examination and hardness testing of each specimen. Heat treatments were performed for one hour at 2600°, 2800°, and 3000°F in vacuum. Figures 17, 18, and 19 show typical grain structures for three of the extrusions.

a. <u>Extrusion A</u>

Figure 17 gives microstructures of the nose and tail sections of Extrusion A at various annealing temperatures. The as-extruded nose section shows only slight deformation of the as-cast grain structure. The as-extruded tail section shows an overall highly worked structure with severe breakdown of the primary as-cast grain. Approximately 14% of the area of the structure is recrystallized. The structure of the extruded sections annealed at 2600°F for one hour indicated approximately 13% recrystallization on the nose and 35% recrystallization on the tail of the extrusion.

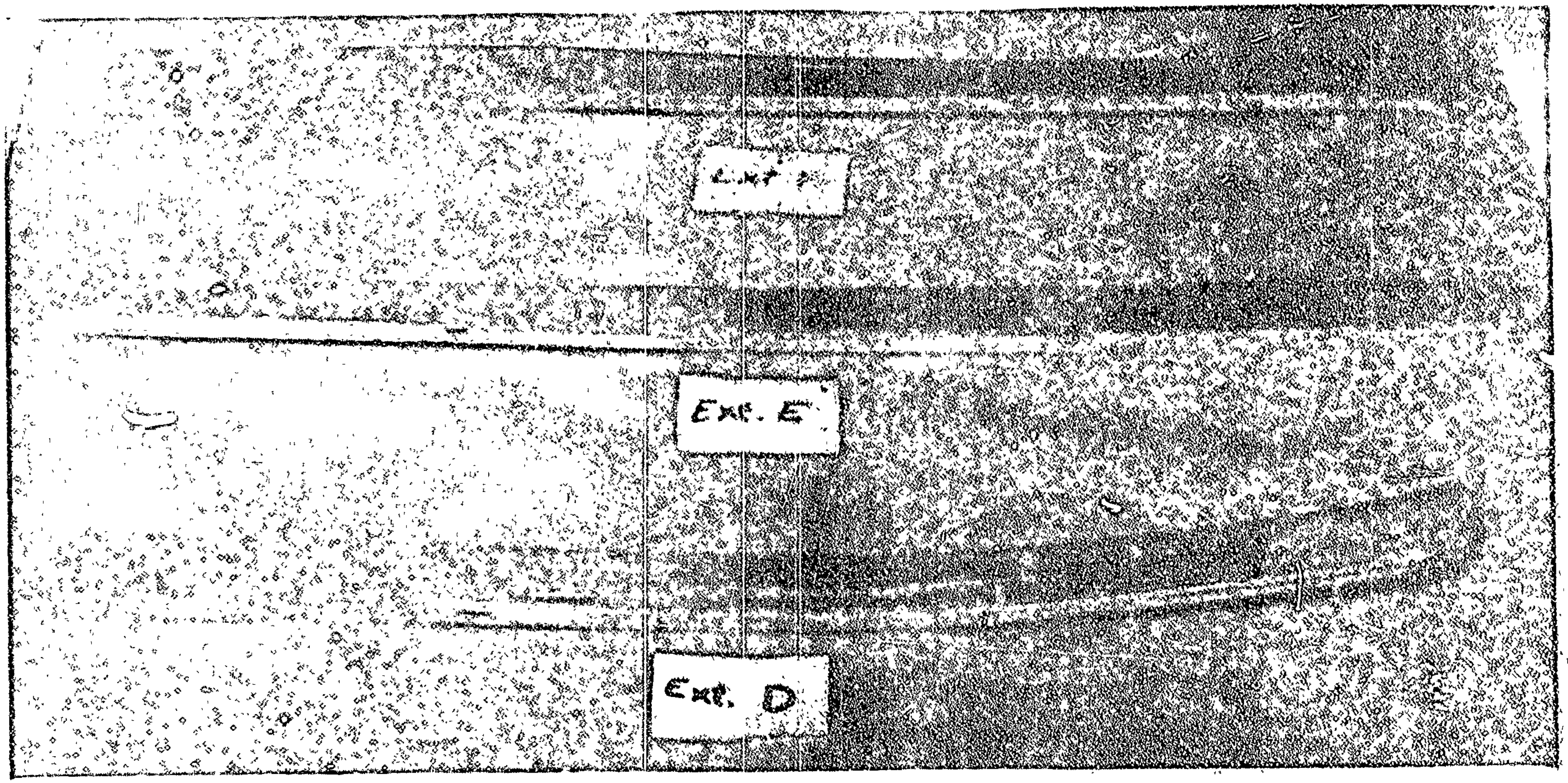


Figure 15

As-Extruded Sheet Bar - From 3.060" Billet
1/2" x 2" x Nominal 36" Long

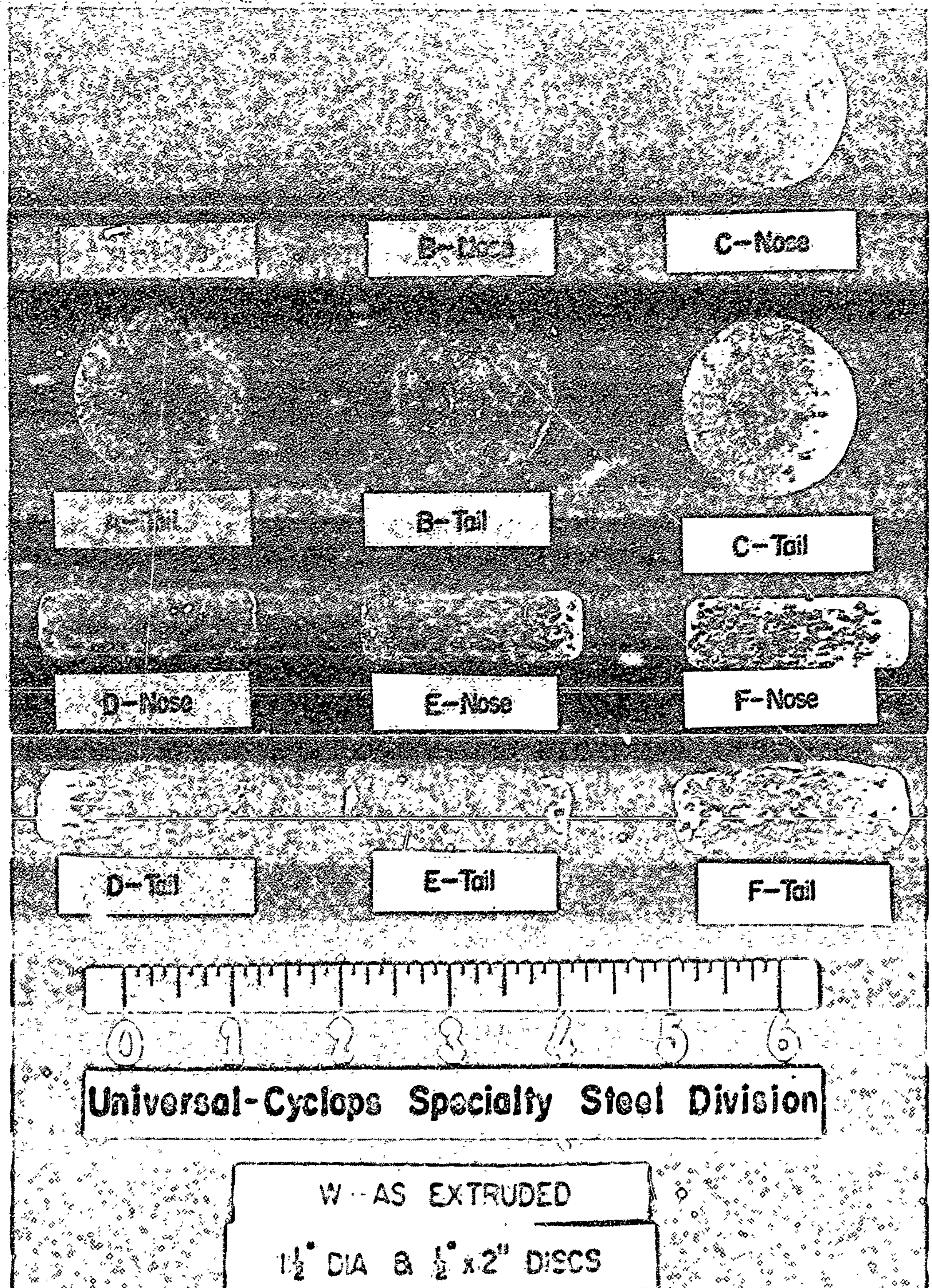
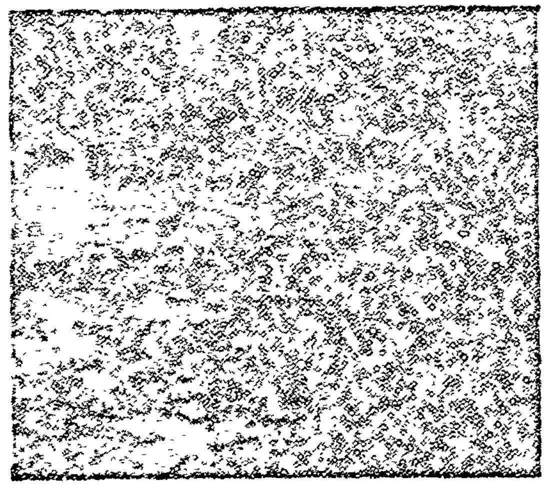


Figure 16

Macrographs of Extrusions

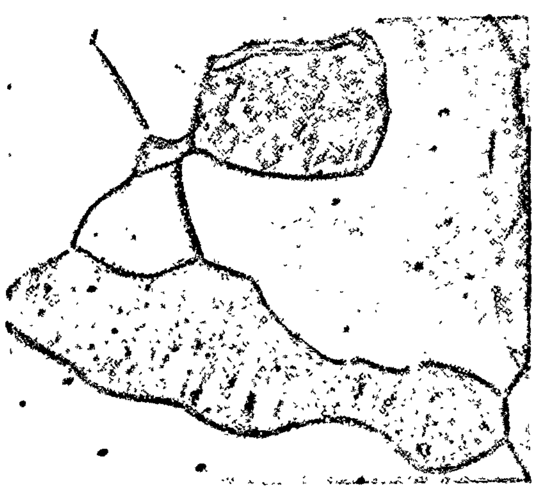
R9773
AN_1 As-Extruded



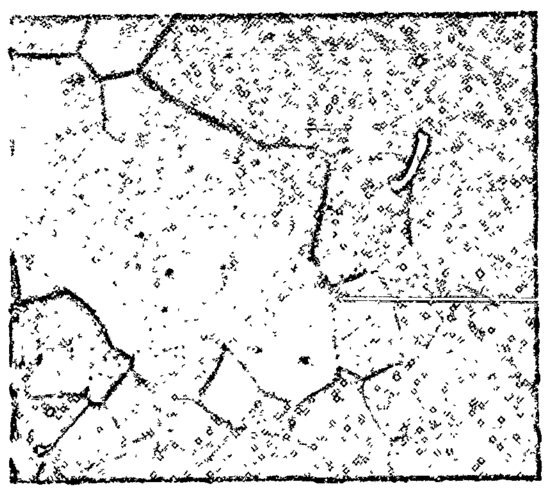
R9777
AT_1 As-Extruded

R9774
AN_2 Annealed 1 Hr/2600°F

R9778
AT_2 Annealed 1 Hr/2600°F



R9776
AN_4 Annealed 1 Hr/3000°F



R9780
AT_4 Annealed 1 Hr/3000°F

Nose | Tail

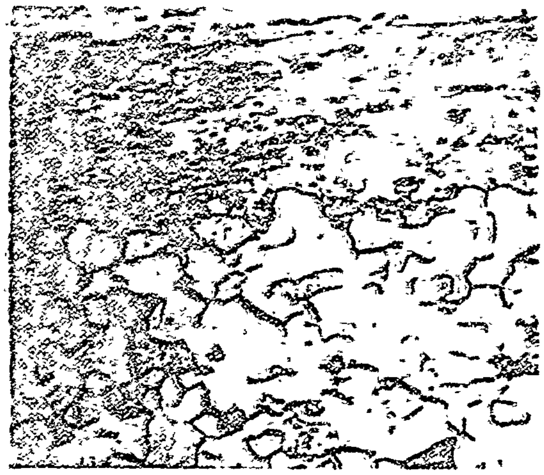
Figure 17

Microstructures of Extrusion A (200X)

The difference in the amount of deformation is readily noted in the two photomicrographs of the nose and tail sections. The nose and tail micrographs annealed one hour at 3000°F indicated complete recrystallization. The phenomena of grain size to degree of work is very evident with the smaller grains in the tail section indicating the results of prior cold work.

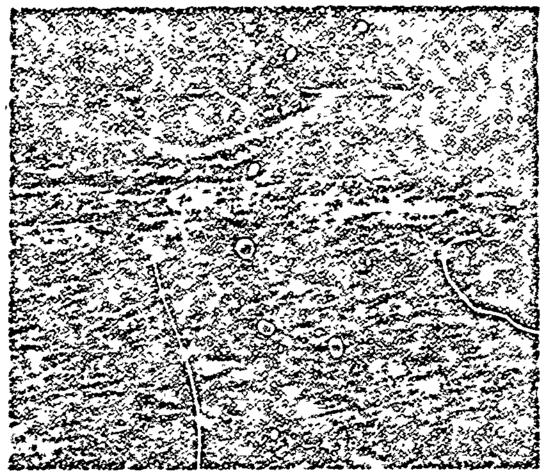
b. Extrusion E

Figure 18 shows photomicrographs of the nose and tail sections of Extrusion E at various annealing temperatures. The nose section of the extruded sheet bar in the as-extruded condition indicated approximately 12% recrystallization, as opposed to 23% recrystallizati on the extruded tail end section. The two as-extruded sections show the increased amo t of wrought structure due to the higher extrusion ratio used on the sheet bar. However, the difference in the amount of work is readily noted between the two microstructures, with the tail micrographs showing a predominately wrought structure with numerous small recrystallized grains. The microstructures of the nose and tail sections annealed one hour at 2600°F indicated 50% and 25% recrystallization respectively. The micrographs show an effect which exists in most of the sample, i.e. the strain energy from extrusion is very non-uniform from grain to grain and in subsequent heat treatments, some grains recrystallize faster than others. The nose microstructure shows wrought structures, stages of initial recrystallization, small equiaxed grains, and coarse grain all adjacent to one another. The tail micrograph shows narrow bands of recrystallization and former primary grain boundaries. The nose and tail sections annealed one hour at 3000°F show massive and uneven grain size with the grain structure of the tail section being considerably smaller due to the prior cold-work history and initial breakdown of the as-cast grain.



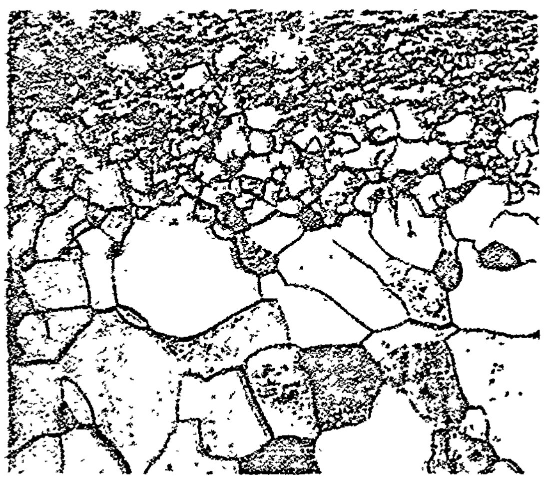
R9783

EN_1 As-Extruded



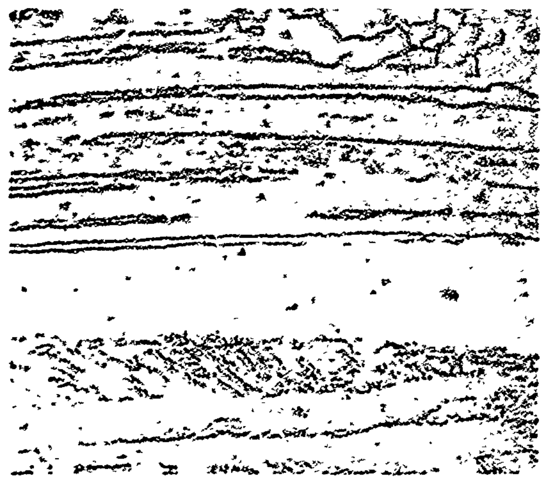
R9787

ET_1 As-Extruded



R9784

EN_2 Annealed 1 Hr/2600°F



R9788

ET_2 Annealed 1 Hr/2600°F

R9786

EN_4 Annealed 1 Hr/3000°F

R9798

ET_4 Annealed 1 Hr/3000°F

<u>Nose</u>

<u>Tail</u>

Figure 18

Microstructures of Extrusion E (200X)

c. Extrusion F

The low magnification of the as-extruded sample, tail section given in Figure 19, reveals the overall extruded structure. Several primary grain boundaries can be observed. Recrystallization is evident within one grain and other areas are shown in the early stages of recrystallization.

Investigation of the overall structure of the six extrusions revealed that:

1. Severe duplex structure was present in the as-extruded condition or after low temperature heat treatment.

2. On the extruded rounds, recrystallization consistently was initiated at the surface and proceeded to the center due to the variance of cold-work from surface to center.

3. In order to completely recrystallize a cross section of either rounds or sheet bar, severe grain growth occurred in areas of early recrystallization.

Figures 20 and 21 are plots of the estimated percent recrystallization of the as-extruded heat treated specimens. In Figure 20, it will be noted that the nose specimens were very slow to recrystallize and only one out of three actually reached 100%. This can probably be attributed to the fact that the samples were cut too close to the extruded nose, which received very little reduction. The tail samples come closer to representing the overall structure. Some correlation with extrusion temperature and speed is evident. Note that "A", extruded at the highest temperature, has the highest rate of recrystallization. "B" and "C" were extruded at the same temperature, however, the extrusion speed of "C" was slower.

R9801

Figure 19

Sample FT_1

Extrusion F - Tail Section

As-Extruded - 50X

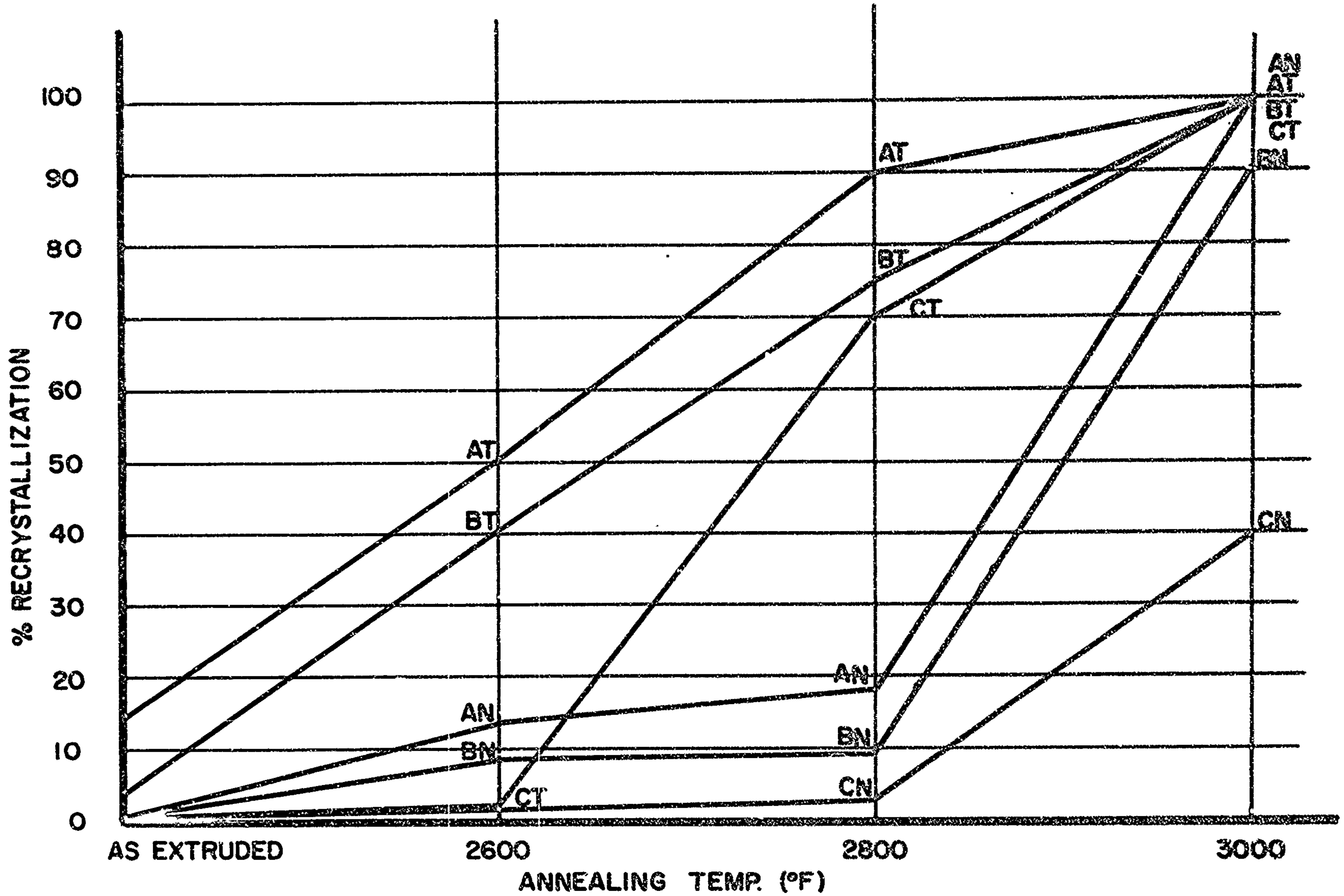


FIGURE 20

ESTIMATED % RECRYSTALLIZATION VS ANNEALING TEMP.—EXTRUDED ROUNDS

Plots of recrystallization of the sheet bar in Figure 21 show no correlation with extrusion temperature. It will be observed, however, that the higher reduction induced in the sheet bar extrusion resulted in consistently lower recrystallization temperatures.

Hardness measurements were taken on all the micro samples. Figure 22 shows a plot of the hardness of the extruded rounds. As on the estimated percentage recrystallization, the tail samples of the three extrusions show good correlation with temperature. After a 3000°F heat treatment, the tail samples are very close in hardness. In Figure 23, the sheet bar hardnesses, as in the case of recrystallization, show no correlation with the extrusion temperature. The average fully annealed hardness of the rounds was 363 DPH as compared to 359 DPH for the sheet bar.

4. <u>Press Forged Extrusion</u>

The three extruded rounds were recrystallized, cropped, and conditioned. They were then press forged to a nominal 3/4" x 2" sheet bar shown in Figure 24. The billets were heated in a 2300°F gas fired furnace and forged on a 1500 ton fast acting press forge. The actual forging temperature ranged from 1925° to 1975°F. Cracks were observed visually on the edges of all three forgings. However, macro slices shown in Figure 25 indicate that the depth of the cracks were very slight.

Hardness measurements on transverse sections from each of the three press forged extrusions are shown in Figure 26. As the starting conditions and the forging parameters were essentially the same for each of the three, the correlation of hardness measurements was expected. Hardness surveys indicated that the center of the forgings have been severely cold-worked. Forging A was more thoroughly investigated and definite metal flow patterns could be

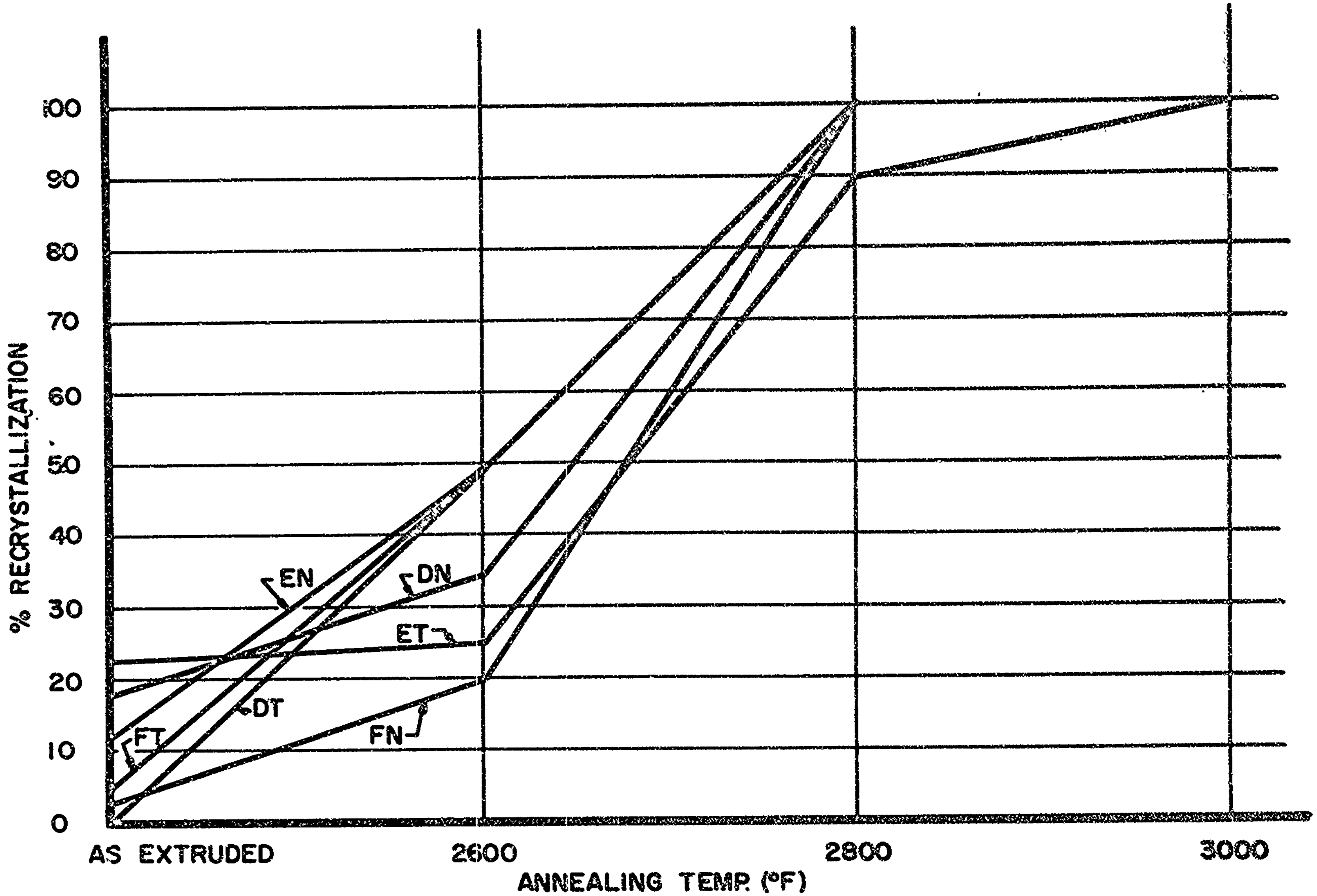


FIGURE 21

ESTIMATED % RECRYSTALLIZATION VS ANNEALING TEMP. — EXTRUDED SHEET BAR

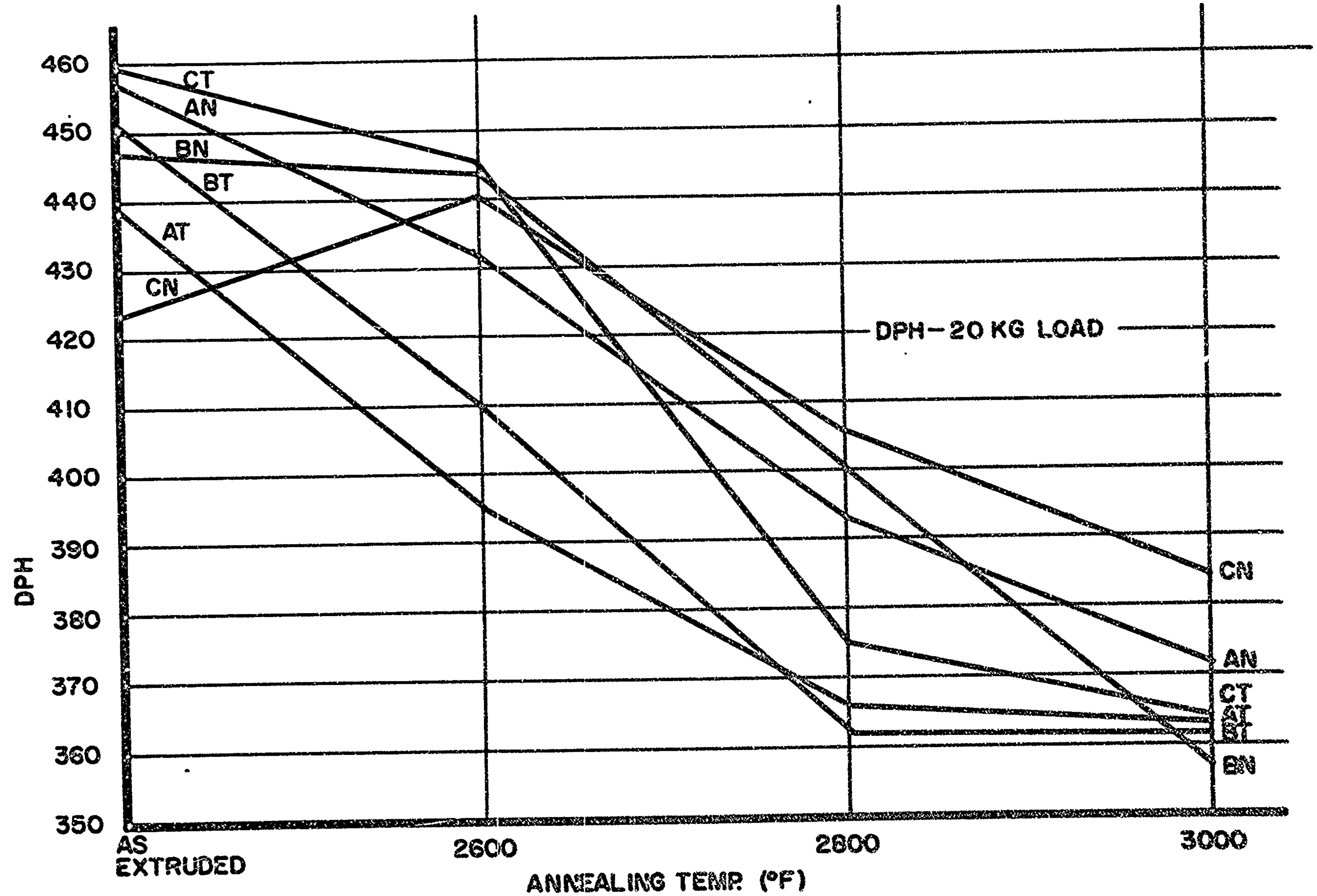


FIGURE 22
HARDNESS VS ANNEALING TEMPERATURE, EXTRUDED ROUNDS

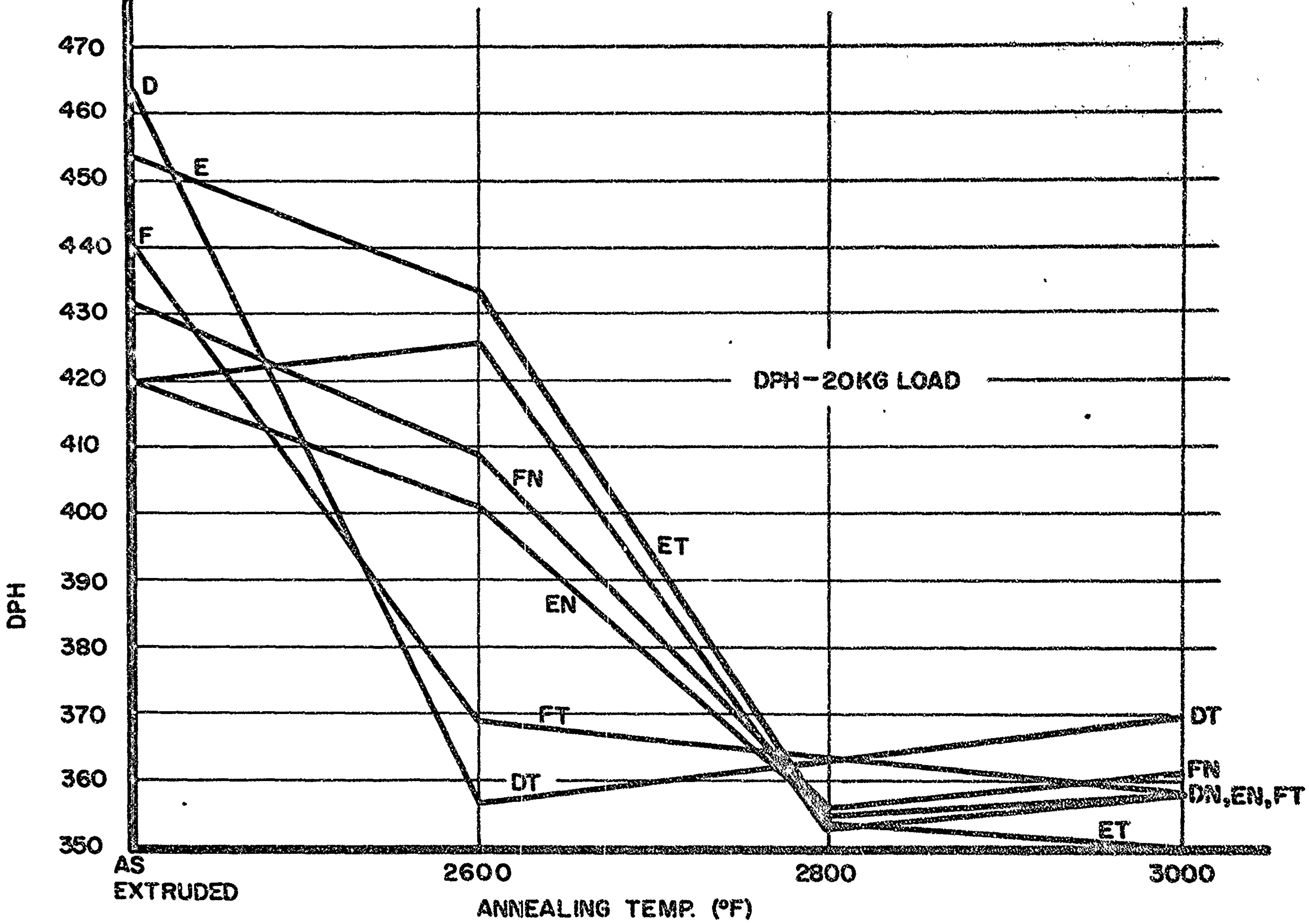


FIGURE 23

HARDNESS VS ANNEALING TEMPERATURE, EXTRUDED SHEET BAR

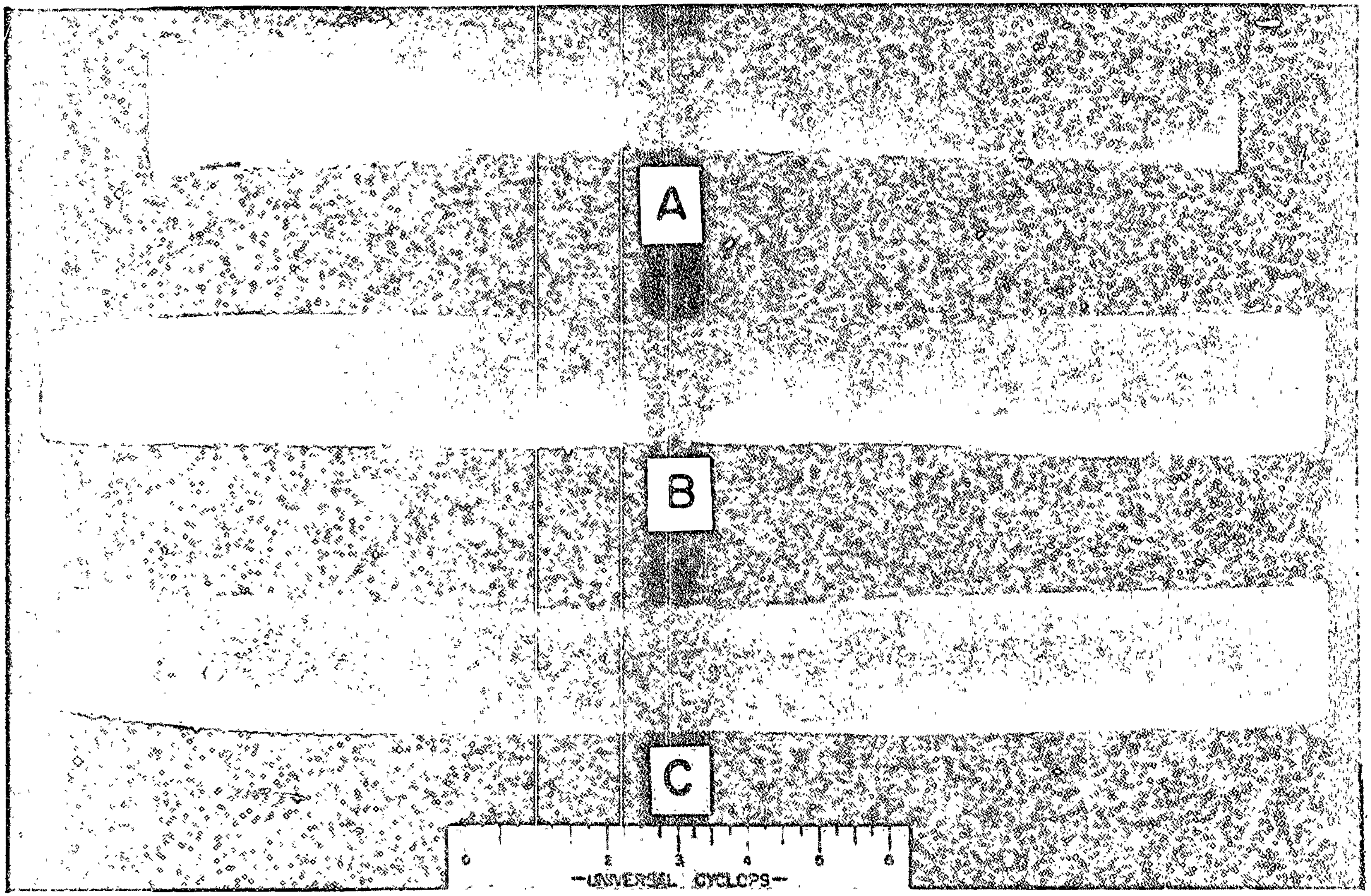


Figure 24

..., Press Forged Extrusions

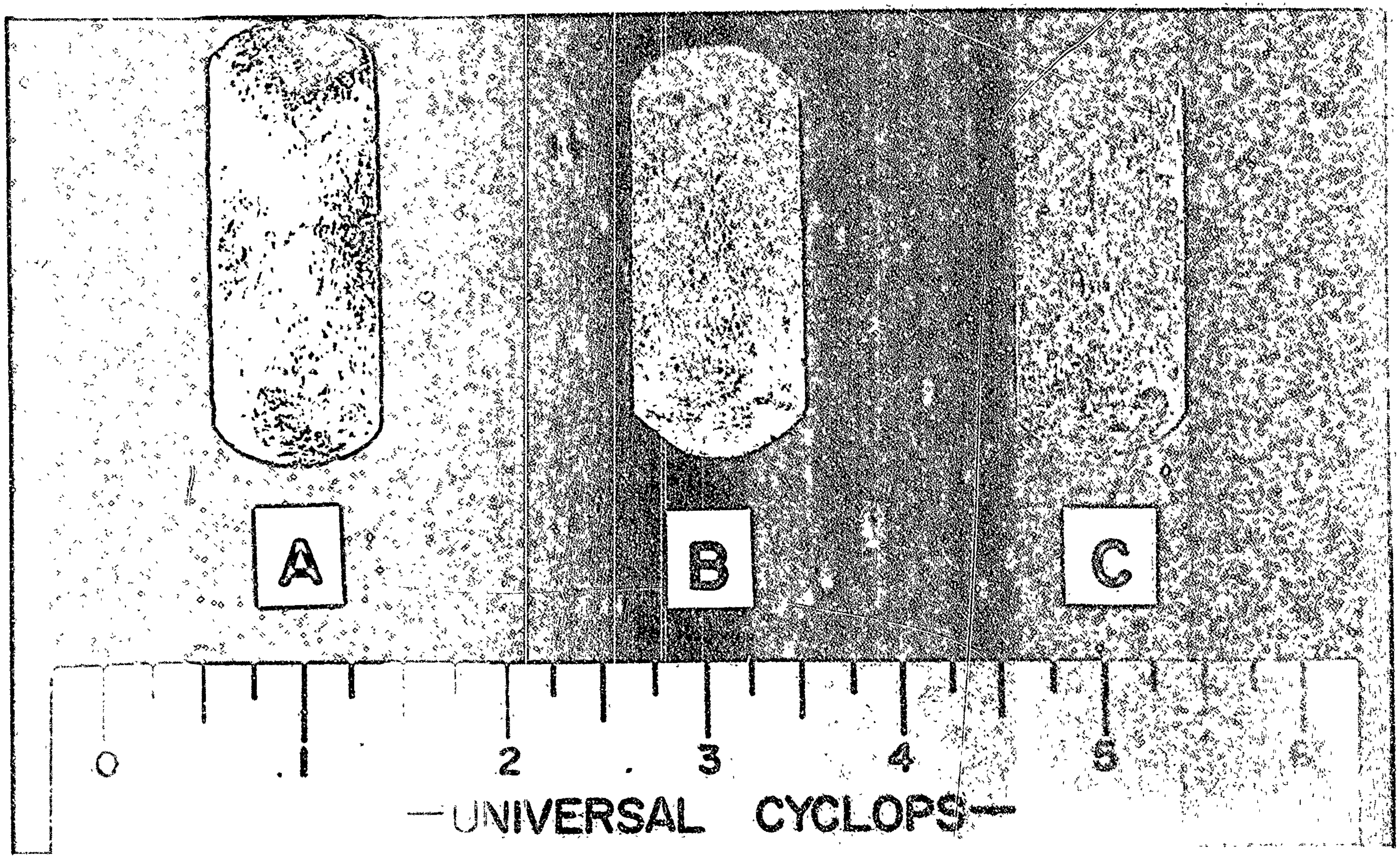


Figure 25

...aphs of Press Forged Extrusions

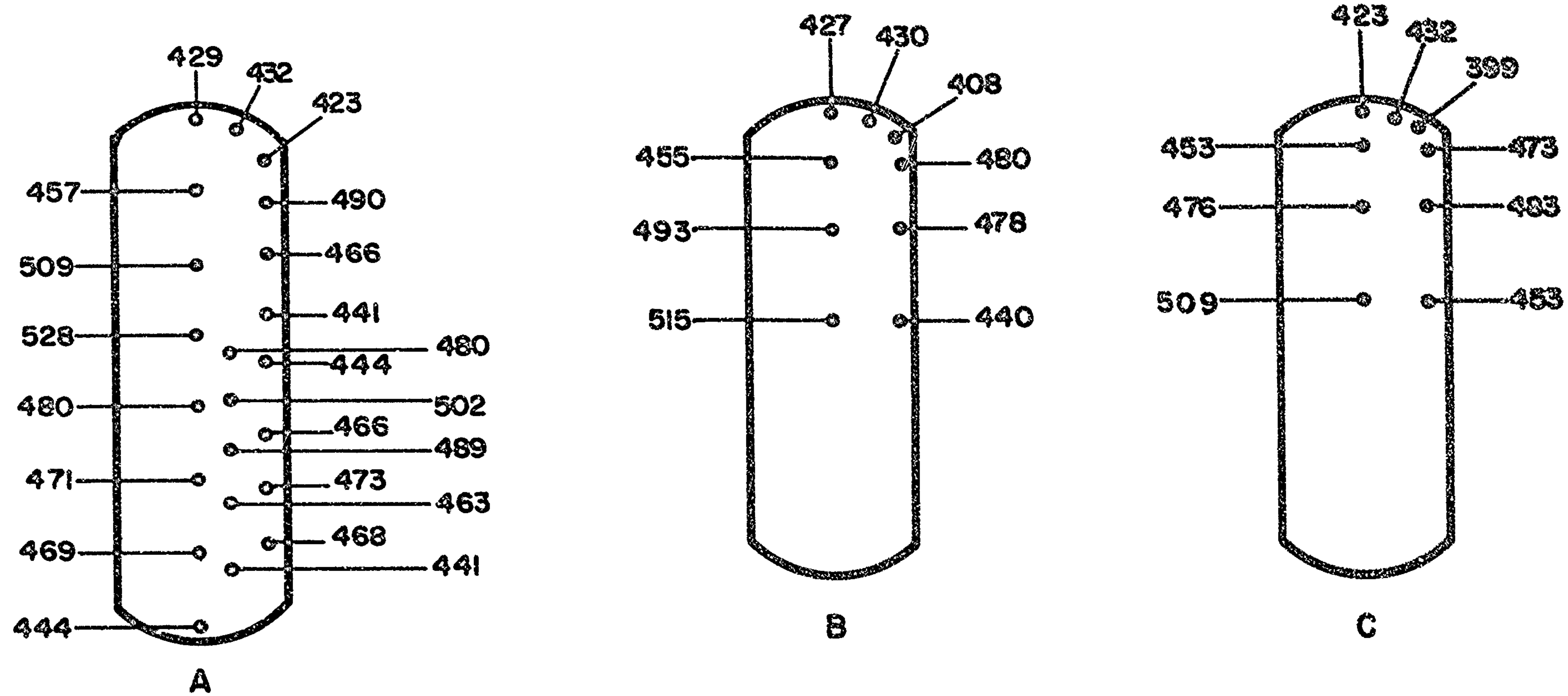


FIGURE 26
HARDNESS OF PRESS FORGED EXTRUSIONS, DIAMOND PYRAMID HARDNESS - 20 KG LOAD

established from the hardness data. The areas of least work were the radius edges not in contact with the die. The next area of minimum work was at a point midway along the flat pressed edges which would be that area receiving initial contact with the die. This point would be in simple compression so that metal flow would be perpendicular to the die face.

Very little conditioning was required to prepare the forgings for rolling. A nominal yield from conditioned extrusion to conditioned forging was 80%.

5. <u>InFab Forging Studies</u>

In order to evaluate the potential of InFab as a forging and rolling source for the ultimate objectives of the program, two 1-1/2" round extrusions were prepared for investigation. These extrusions were ultrasonically inspected and threaded on one end to facilitate holding during impact forging. The bars were forged individually to nominal 1/2" thick sheet bar according to the schedule given in Table XV.

TABLE XV

INFAB FORGING SCHEDULE

Identification	Reheat	Temperature °F Heating	Temperature °F Initial Impact	Temperature °F Final Impact	Thickness
KC1088-A	0	3980	3325	2650	3/4"
KC1088-A	1	3825	3200	2500	1/2"
KC1088-B	0	4075	3400	2700	7/8"
KC1088-B	1	3860	3150	2500	5/8"
KC1088-B	2	3780	2975	2450	3/8"-1/2"

Although heating temperatures in the range of 4000°F were utilized, rapid heat loss due to the small mass and high thermal conductivity resulted in lower forging temperatures

than anticipated. The only crack observed was a small burst which extended approximately 1/2" into the bottom of one piece. The forgings had an irregular cross section which was attributed to excessive reductions per impact. However, this was required on these pieces to minimize reheating. Since one forging was considerably smaller than the other, they were sectioned to provide one sheet bar and three sheet bars respectively, for subsequent rolling studies, which will be covered later in this report.

C. Scale-Up to 4" Diameter Conditioned Ingot

In order to meet the Phase III objective of the program, the production of 24" x 24" sheet product, it was necessary to establish an ingot breakdown process for 4" diameter conditioned ingot. The results on preliminary investigations on ingot breakdown indicated that although direct press forging, with proper control of processing variables, could be utilized as an initial breakdown method, the technology developed in extrusion of the as-cast ingot was more advanced and offered the best chance for success and subsequent scale-up. Since extrusion to rounds followed by press forging to sheet bar and direct extrusion to sheet bar were accomplished successfully in the initial ingot breakdown evaluation from 3" diameter ingot, both methods were investigated in the breakdown of 4" diameter conditioned ingot.

1. Procedure

The scale-up of the extrusion of 4" diameter conditioned ingot required larger extrusion facilities than had been used previously. The DuPont 2750 ton press was selected for future extrusion investigations based upon their capability to closely duplicate the temperatures, extrusion speeds, and other extrusion characteristics utilized in the initial evaluation. In addition, the size capability of the press allowed for the extrusion of 8" diameter ingots which coincided with the end requirements of the program.

The use of different extrusion facilities, in addition to larger billets, involved several unknown variables, such as the effect of increased transfer time from furnace to press and throttle settings to produce the desired speed of 8" to 12" per second. Because of these variables, one billet was extruded as a pilot evaluation prior to extruding the remaining billets on the program.

The first billet was heated to 3050°F in argon atmosphere at a rate of 50° to 75°F per minute to 2000°F and 150° to 200°F per minute to 3050°F. The billet was removed from the furnace and moved to the press using an automatic transfer device. During this transfer, the billet was rolled over pyrex glass wool mat containing a powdered 3 KBA glass and this coating provided the lubrication for extrusion. After extrusion, the billet was immediately transferred to a furnace and annealed for one hour at 2500°F and then slow cooled in vermiculite.

2. <u>Extrusion Parameters</u>

The pilot ingot was extruded from 4" diameter conditioned ingot to 2" diameter billet or a 4:1 extrusion ratio under the following conditions: Transfer time, 50 seconds; extrusion speed, 4.5" per second; breakthrough pressure, 124,000 psi; running pressure, 124,000 psi.

3. <u>Extrusion Evaluation</u>

Visual observation of the extrusion after annealing and slow cooling in vermiculite showed several slight surface tears. [illegible] burst of approximately 1" and a relatively deep tail pipe of approximately 4" were also observed. Contact ultrasonic inspection [illegible] the extrusion to be sound; however, immersion ultrasonic [illegible] indicated a longitudinal crack from the surface to mid-[illegible] extending almost the entire extrusion length. Samples

were taken for hardness and micro examination. Even though this extrusion had been annealed, the average hardness of 465 DPH was 10 DPH higher than that shown for as-extruded hardness in the initial breakdown of the 3" diameter ingots. Considering the stress relief treatment, the hardness was 25 to 30 DPH higher than expected. Observation of the microstructure showed complete cold-work at the nose and 5% to 10% recrystallization in the tail. The previous extrusions with a corresponding heat treatment had shown 5% to 10% recrystallization in the nose and 25% to 30% in the tail. It was concluded that the actual extrusion temperature was considerably lower than desired. Although the furnace temperature was the same as that previously utilized, the longer transfer time and slower extrusion speed apparently resulted in the lower extrusion temperature.

From the results of the pilot evaluation, four additional billets were extruded at increased furnace temperature and increased throttle settings to provide increased extrusion speeds. Extrusion data for these billets is shown in Table XVI. It can be noted that the 200°F increase in temperature over the pilot billet caused a considerable drop in extrusion pressure even though the speed was increased. The extrusion speed of 10" per second was within the desired range of 8" to 12" per second. The fifth billet, extruded at 3400°F, did not result in lower pressures or higher speeds as expected. The 2" diameter extrusions are shown in Figure 27. The cracking problem noted in the pilot extrusion persisted in the remaining four. However, these cracks were not as severe and sound material was salvaged for further processing. Three additional billets were extruded after evaluation of this material and the extrusion data for these is also incorporated in Table XVI. The higher temperature utilized resulted in improved yields. As shown, one of these billets was extruded directly to

TABLE XVI

EXTRUSION DATA FOR 4" DIAMETER BILLETS

Ident.	Weight	Furnace Temperature °F	Pressure psi Breakthrough	Pressure psi Running	Speed (IPS)	Evaluation	% Yield
KC1135	91	3050	124,000	124,000	4.5	Severe Longitudinal Cracks	0
KC1151	80	3200	113,000	98,000	10	Longitudinal Crack On Trailing 20".	31
KC1158	82	3200	113,000	113,000	10	Longitudinal Crack On Trailing 10".	62
KC1160	81	3200	113,000	113,000	10	Normal Nose and Tail Loss Only	89
KC1161	64	3400	113,000	95,000	10.5	Intermittent Cracks Along Length.	0
KC1175	84	3500	108,000	100,000	10.5	Normal Nose and Tail Loss	89
KC1176	81	3500	108,000	95,000	10	Normal Nose and Tail Loss	86
*KC1178	59	3500	108,000	100,000	10	Normal Nose and Tail Loss	87

Constant Reduction Ratio - 4:1

Constant Extrusion Size - 2" Diameter

*Extruded to 1" x 3" Rather Than 2" Diameter

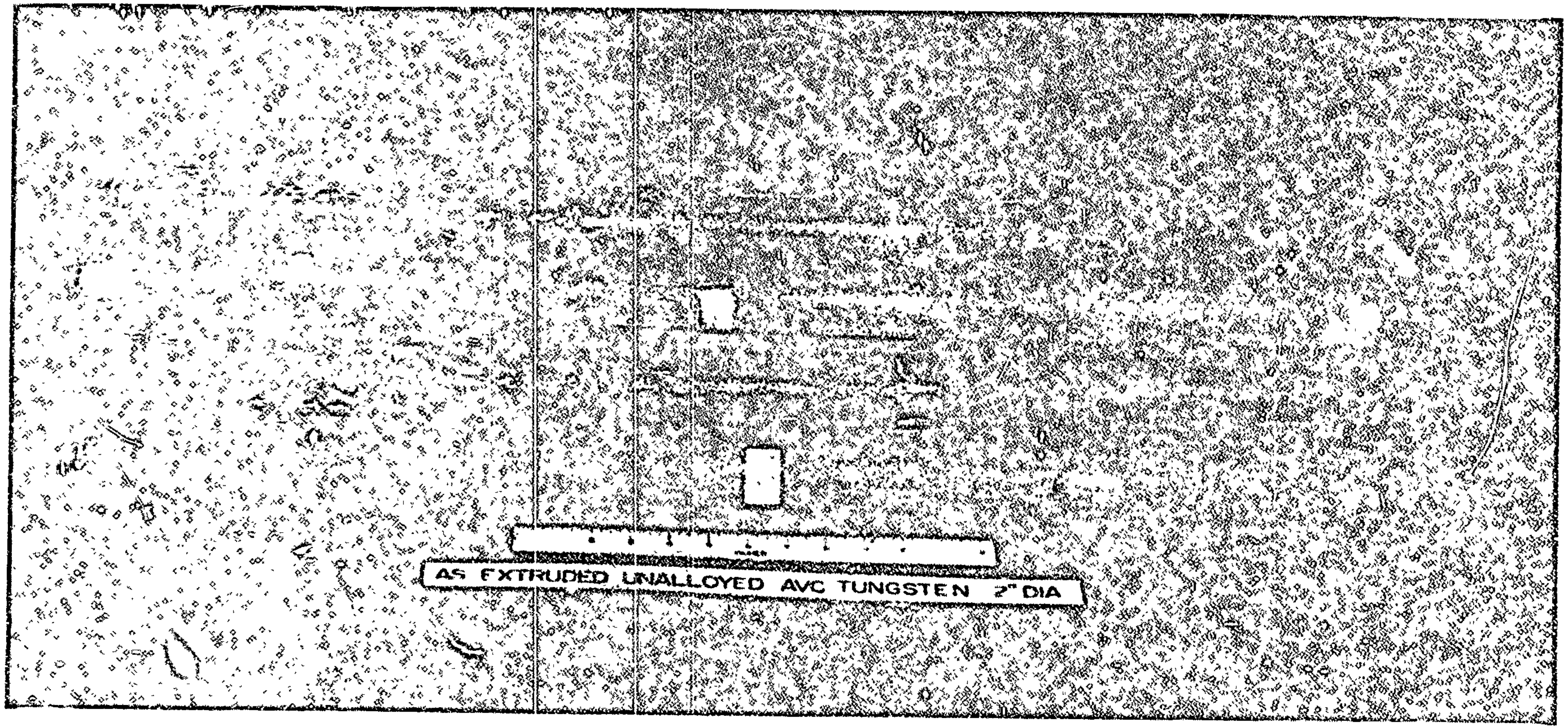


Figure 27

2" Diameter As-Extruded AVC Tungsten

1" x 3" sheet bar. The pressure required for the sheet bar was the same as that for rounds and the resultant product was very satisfactory.

4. Press Forged Extrusions

From 2" diameter cropped and conditioned extrusions, 10" mults were cut for forging evaluation. Forging was accomplished utilizing a 2700°F hydrogen atmosphere furnace and six pieces were press forged to a nominal 1" x 3" x 10" sheet bar. The actual forging temperature was 1800° to 2100°F. The relatively large difference between heating and forging temperatures is attributed to transfer time and the large surface to volume ratio which resulted in rapid radiant heat loss. All forgings were annealed for one hour at 2800°F and slow cooled in vermiculite and sandblasted. Minor surface cracking was noted in all forgings; however, the yield loss on forging was limited to approximately 8%.

5. Summary

The results of extrusion from 4" diameter ingot directly to 1" x 3" sheet bar indicates comparable yield results to the extruded 2" diameter rounds. Since there is an 8% yield loss in press forging 2" diameter round to sheet bar, it was decided that the improved yields dictated the use of extruded sheet bar for the final pilot production run.

D. Scale-Up of Extrusion for 6" Diameter Conditioned Ingot

1. Extrusion Evaluation

Four 6" diameter conditioned ingots were extruded on the DuPont 2750 ton press in order to scale-up to the Phase IV requirements of the program which called for 36" x 36" sheet product. Two of the conditioned ingots were extruded to 3" diameter rounds for subsequent press forging to sheet bar and the other two were

extruded directly to 1-3/4" x 4" cross section sheet bar. Table XVII lists the extrusion parameters used and the resultant pressure requirements. As shown, all billets were extruded at a temperature of 3200°F (furnace temperature 3500°F) based on the successful results of 4" diameter billets extruded at this temperature. The breakthrough pressure requirements were relatively consistent except for the first extrusion which was somewhat higher. It is shown by running pressure that the sheet bar extrusions require slightly more pressure than the rounds.

The average extrusion constant of the previous 4" billets was 81,600 psi. This is lower than the first billet but higher than the other three. Figure 28 shows the two as-extruded rounds after sandblasting. Also indicated are the end cropping requirements determined by contact ultrasonic inspection. The relatively large amount to be cropped from the tail (nominal 4") is due to a deep tail pipe that actually does not represent a complete solid piece since this area is hollow. The picture shows that the general surface was excellent and it should also be noted that no die wash occurred. Actually both billets were put through the same die which had not been accomplished on previous runs. An as-extruded sheet bar is also shown in Figure 28. This photograph also indicates that nc die wash occurred which is remarkable considering the sharp corner angles required on the sheet bar die. The fact that no die wash occurred on these extrusions is further verified by the physical dimensions shown in Table XVIII.

TABLE XVII

EXTRUSION DATA FOR 6" DIAMETER BILLETS

Heat Number	Billet Weight	Billet Shape	Temperature (°F)		Pressure (psi)		Speed (IPS)	Extrusion Constant (K)
			Shaw	Optical	Breakthrough	Running		
KD1147	231	Round	3200	2680	118,000	97,000	16.5	85,000
KD1148	233	Sheet Bar	3200	2665	108,000	104,000	16	78,000
KD1167	303	Round	3200	2675	100,000	93,000	17	72,250
KD1168	153	Sheet Bar	3200	2705	108,000	105,000	18	78,000

Extrusion Constant $K = \dfrac{P}{A \ln \frac{A}{a}}$; Where: P = Maximum Pressure in Pounds
K = Extrusion Constant in psi
A = Cross Sectional Area of Container
a = Cross Sectional Area of Extrusion

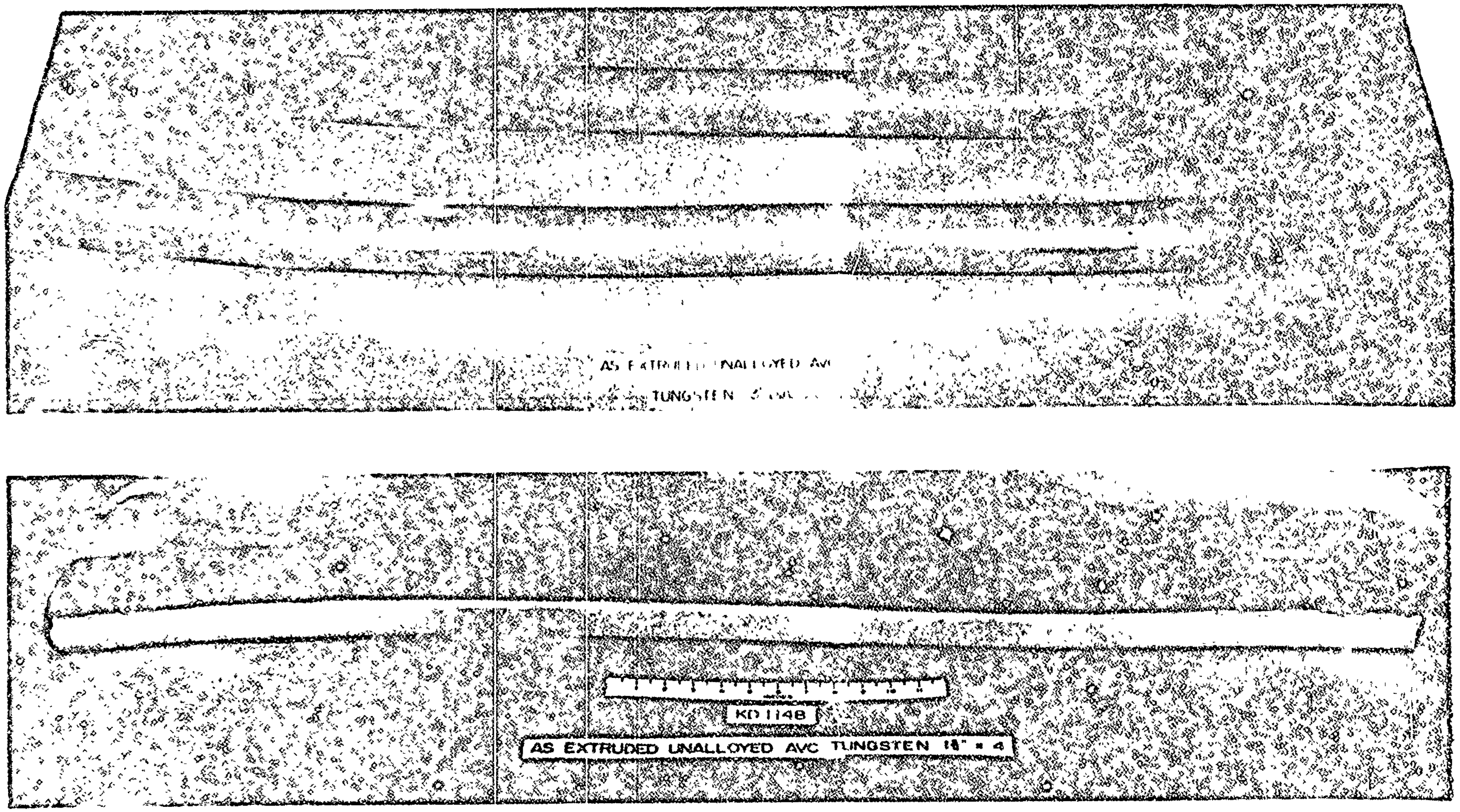


Figure 28

As-Extruded 3" Diameter Rounds and
1-3/4" x 4" Sheet Bar From 6" Diameter Conditioned Ingots

TABLE XVIII

PHYSICAL DIMENSIONS OF EXTRUSIONS

Heat Number	Nose	Tail	As-Extruded Length
KD1147	3.034" Ø	3.018" Ø	48"
KD1148	3.984" x 1.767"	3.990" x 1.765"	49"
KD1167	3.043" Ø	3.030" Ø	59"
KD1168	3.975" x 1.765"	3.975" x 1.768"	32"

To more accurately evaluate the internal quality of the round extrusions, immersion ultrasonic evaluation was required. In order to accomplish this, straightening was necessary. The extrusions were heated to 2300°F in a hydrogen atmosphere furnace, straightened on a 1500 ton press and subsequently reheated to 2300°F, soaked for ten minutes and then buried in vermiculite. After cooling, immersion ultrasonic examination indicated that on both extrusions a longitudinal crack varying in depth up to 1/2" extended along the entire length. However, the results were not precise due to slight surface defects. In addition, 12" on the trailing edge of heat KD1167 appeared to be cracked from surface to center. Since the surface defects were preventing an accurate evaluation, the extrusions were machined to 2.850" and then surface ground to 2.830" diameter. They were immersion ultrasonically examined again and the results plotted in Figure 29. From this examination, heat KD1167 was free of defects except for 2.5" on the nose end. On heat KD1167, 12" on the trailing end was cracked, tapering from surface to center. In addition, 11" in the center of the extrusion was cracked to a maximum depth of 1/4", and 1" on the nose was cracked to a depth of 1/2". As indicated in the diagram, two forging mults were cropped from each extrusion. The cracked areas on heat KD1167 were ground out prior to forging.

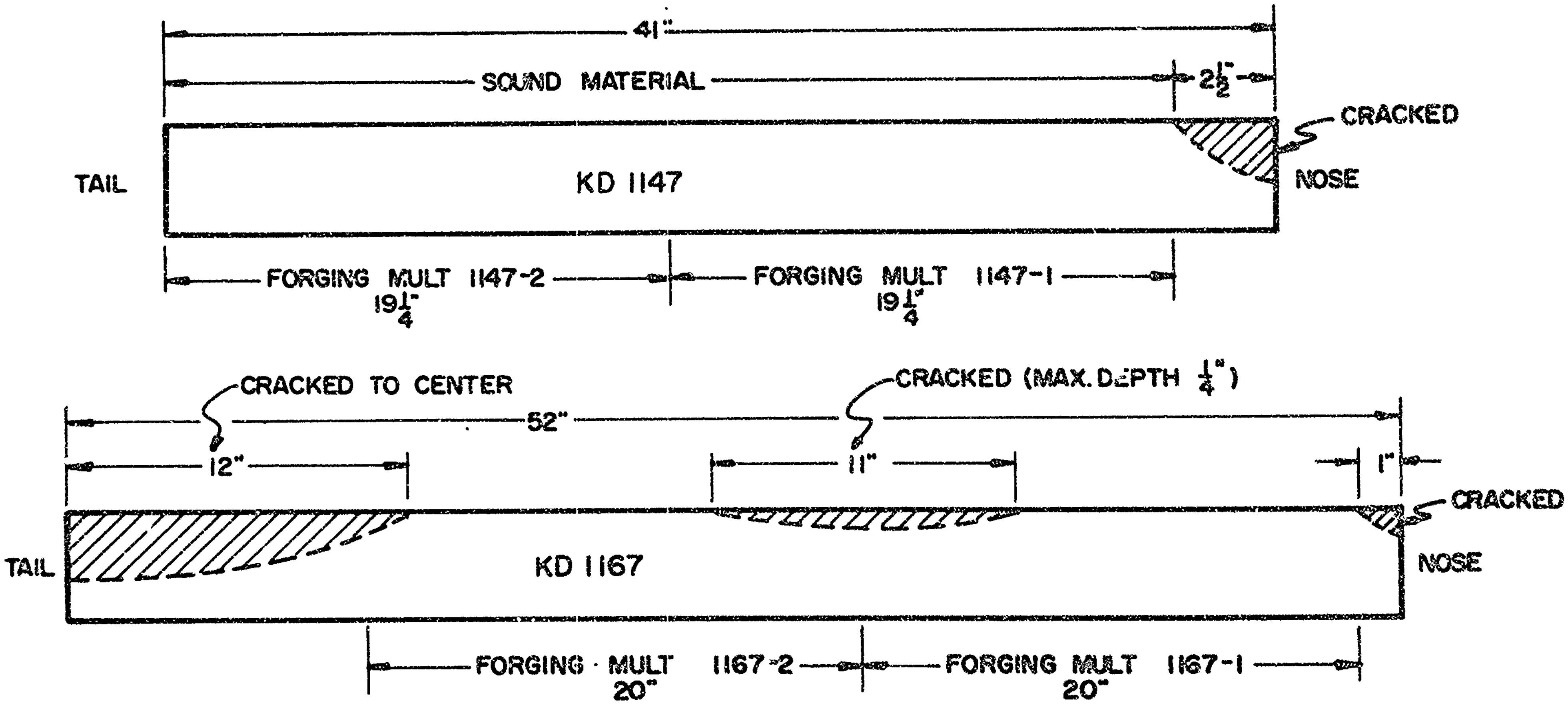


FIGURE 29

ULTRASONIC EVALUATION OF EXTRUDED ROUNDS

After cropping the as-extruded sheet bar, minor surface conditioning was required to prepare it for subsequent rolling. This extrusion was also cropped into two mults for the rolling operation.

2. Press Forged Evaluation

For the forging operation on the four 3" diameter extruded mults, a 1500 ton hydraulic press was used in conjunction with a 3000°F hydrogen atmosphere furnace. As this press was relatively slow acting, the pieces were heated to 2600°F in order to maintain a nominal 2000°F forging temperature. The actual forging process is as follows:

1. Charge forging mult into 2600°F furnace.
2. Soak five minutes after reaching temperature.
3. Transfer to press and forge 3/4" flats.
4. Reheat to temperature and hold five minutes.
5. Transfer to press, rotate 90° to initial forging direction, and forge to nominal 2" thick.
6. Reheat to temperature, hold ten minutes, discharge and bury in vermiculite.

In Step 5 above, the press stalled out at a 2-3/4" thickness. The first mult was reheated to temperature and an attempt made to forge it down to 2". However, only 1/16" additional reduction was achieved. The remaining three pieces were only forged once in Step 5. After slow cooling, the pieces were sandblasted for inspection. The four mults are shown in Figure 30. Close inspection showed light surface ruptures on all the pieces. Two of the pieces had one larger crack running parallel to the extrusion direction. These cracks in both mults were conditioned

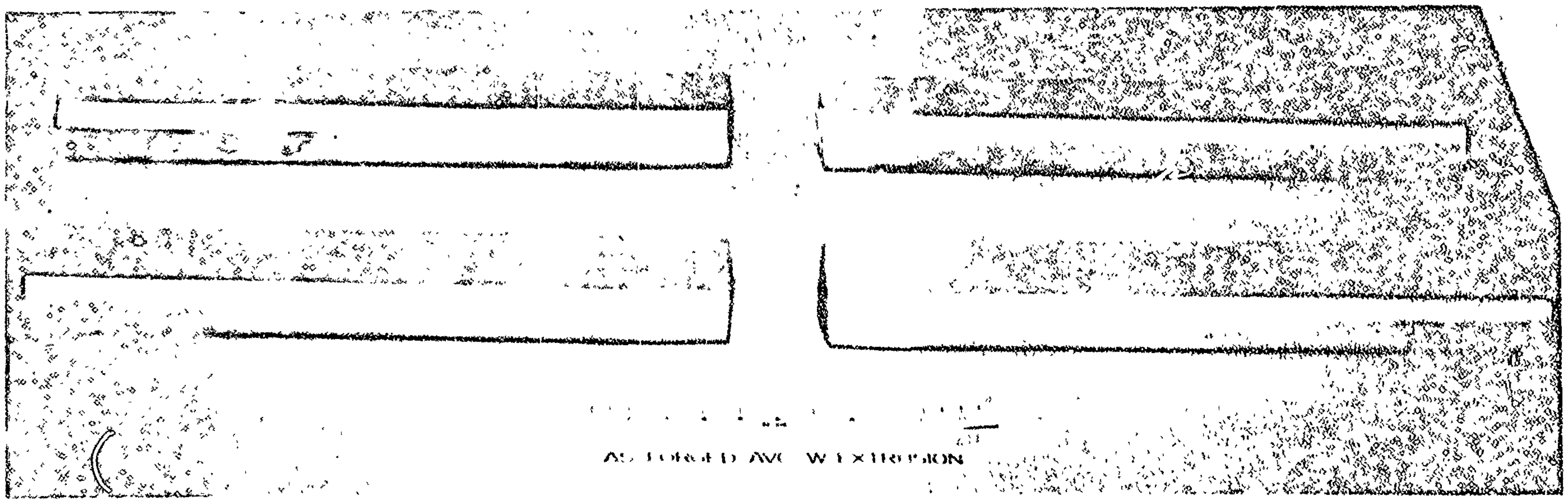


Figure 30

Press Forged Sheet Bar From Extruded 3" Diameter Rounds

out at a depth of 1/8". The remaining surface of these two and the remaining two were ground lightly to remove the light surface ruptures. Yield losses on extrusion and forging are presented in Table XIX.

TABLE XIX

YIELD SUMMARY FROM EXTRUSION BILLET TO SHEET BAR

	Heat Number			
	KD1147	KD1148*	KD1167	KD1168*
Extruded Weight (Lbs)	231	233	301	153
End Loss (Lbs)	23-1/4	25	72	27-3/4
Surface Loss (Lbs)	33-1/2	12	35	10
Forging Mult Weight (Lbs)	168	--	180-1/2**	--
Conditioned Sheet Bar (Lbs)	145	192**	160-1/2**	--
Percent Yield Extrusion to Sheet Bar	62.8%	82.5%	53.3%	75.3%

*Extruded Directly to Sheet Bar
**Two Pieces

It can be observed immediately from the table that the yield in extruding directly to sheet bar is significantly higher and, in addition, eliminates a forging step. The low yield in heat KD1167 is due in part to the cracked portions of the extrusion which, in the table, are included as end losses.

E. Scale-Up of Extrusion for 8" Diameter Conditioned Ingot

The Phase V requirements of the program call for a production run of 36" x 96" sheet at gauges of .020", .040", and .063". In order to meet these requirements and based on previous experiences in extrusion, it was necessary to extrude 8" diameter conditioned ingot directly to sheet bar. The reduction ratio which had proven most desirable throughout the program was 4:1. The

extrusion pressure required for the 6" conditioned ingot in the Phase IV effort indicated that the DuPont press capacity was marginal with respect to extruding 8" conditioned ingot to the same reduction ratio. DuPont personnel recommended that a lower reduction ratio be utilized and it was agreed to attempt a 3.35:1 ratio. A sheet bar die equivalent to this ratio was produced having a 2-1/2" x 6" opening. The billet was extruded using the parameters established for the 6" diameter conditioned ingot. However, the pressures were significantly lower than anticipated. Table XX gives a comparison of the average 6" diameter pressure requirements with that for the 8" diameter.

TABLE XX

COMPARATIVE EXTRUSION PRESSURES

	Pressure (psi)		Extrusion Constant
Billet Diameter	Breakthrough	Running	(K)*
6" Average	108,500	99,750	78,400
8" Average	74,000	72,000	61,200

*Extrusion Constant Calculated Only on Breakthrough Pressure

Since the reduction ratio for the 8" diameter conditioned ingot was considerably lower, the pressure would be expected to be lower. However, the extrusion constant (K) eliminates this variable. Based on the extrusion constant for the 8" diameter ingot at a 4:1 reduction ratio, the breakthrough pressure would have been 84,800 psi or 77.5% of the 109,500 psi available in the 8" container. This data would indicate that a 4:1 reduction ratio is well within the press limitation on an 8" diameter ingot. Visual observation of the extruded sheet bar after sandblasting revealed severe grain boundary tears over the entire surface. Figure 31 shows the two sides of the sheet bar and the surface tears are

Figure 31

As-Extruded Sheet Bar - 2-1/2" x 6"

readily visible. This problem was directly attributed to the low reduction ratio utilized. Extrusion investigations by DuPont confirm the fact that at the lower reduction ratios, tungsten as well as other refractory metals frequently incur severe grain boundary rupture.

The extrusion was stress relieved for one hour at 2800°F and slow cooled in vermiculite. Ultrasonic examination was attempted. However, the rough surface prevented an accurate measure of internal soundness. The nose and tail cropping requirements were determined by this ultrasonic examination and these were abrasively cut. The extrusion was then heated to 2400°F and straightened on a 1500 ton press forge. Using a planer, the two flat surfaces were conditioned from the starting 2-1/2" thickness down to a nominal 2-1/8" thickness which removed the majority of the defects leaving a few which could be hand ground. Ultrasonic examination at this stage revealed a crack approximately 12" long by 1/2" deep on the flat surface running longitudinal to the extrusion direction. The extrusion was spot conditioned and cut to provide the maximum useable quantity for subsequent rolling.

Rolling studies performed on the initial sheet bar extrusion, discussed later in this report, indicated problems associated with the larger grain size of the extrusion which resulted from the low extrusion reduction ratio on the sheet bar. For the second extrusion, the reduction ratio was increased from 3.35:1 to 4.25:1. In addition, the die was designed to provide tapered edges to facilitate initial rolling within the mill capacity. The sheet bar configuration is shown in Figure 32. With the increased reduction ratio, the extrusion temperature was increased to 3500°F in order to be well within the overall press capacity. The billet was extruded at 11" per second with a resulting pressure of 75,000 psi which corresponds to an extrusion constant (K) of 51,800.

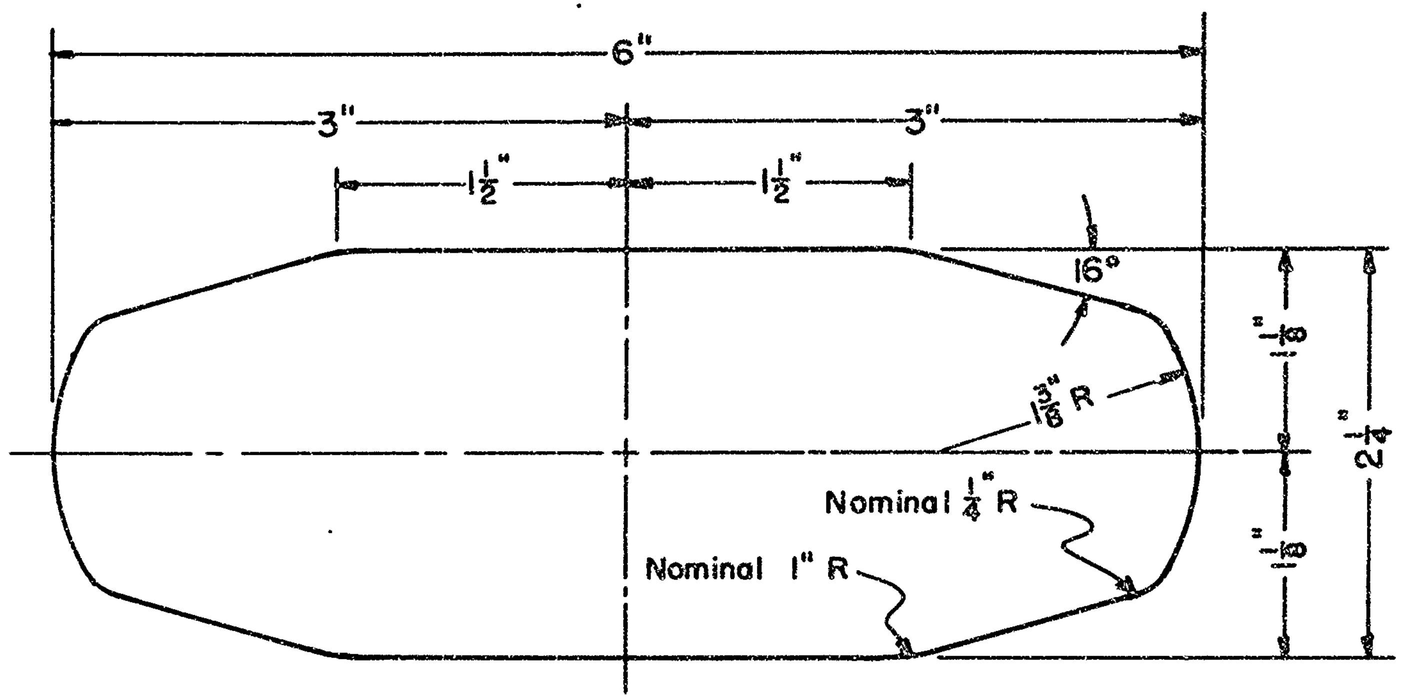
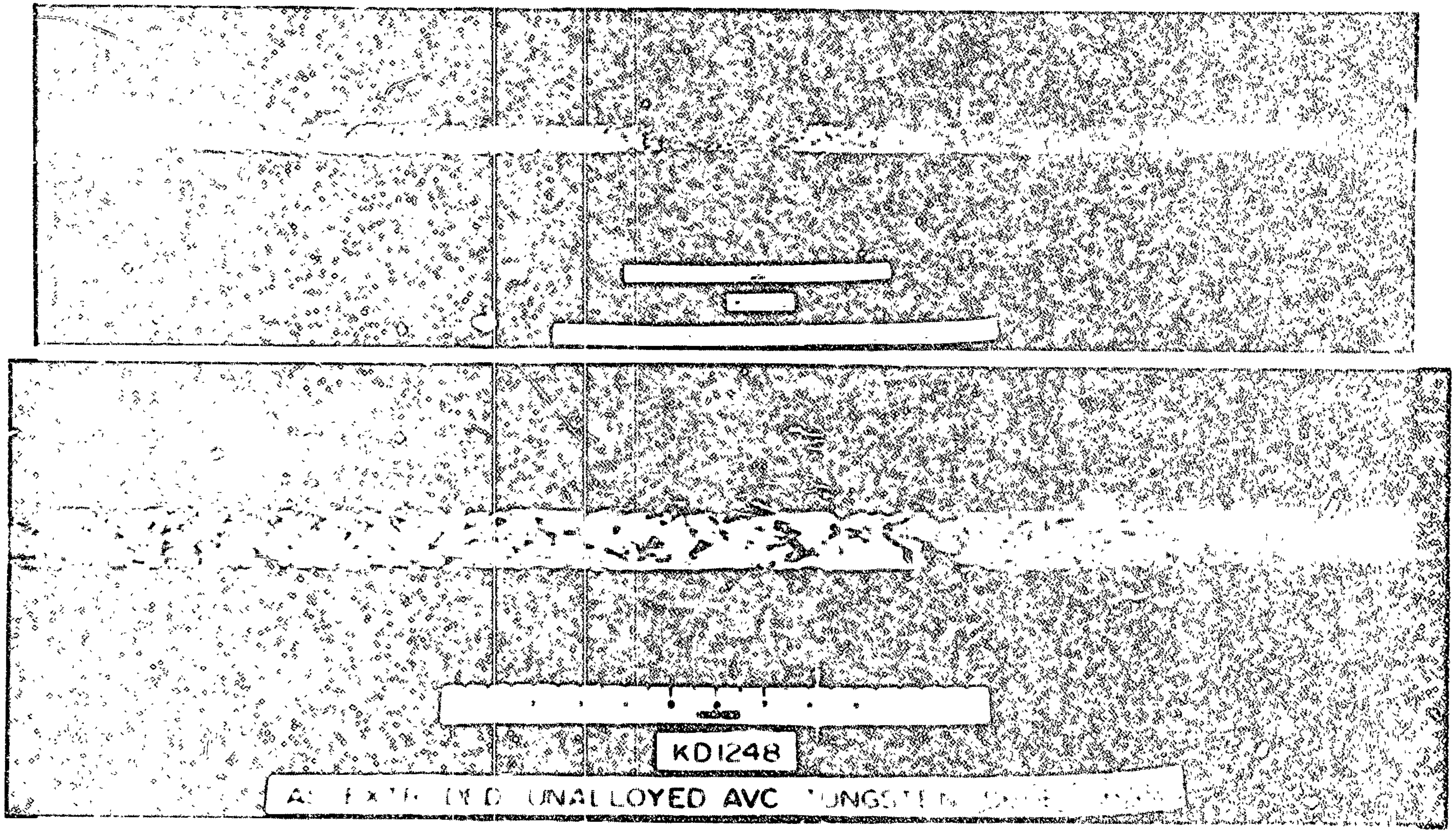


FIGURE 32
EXTRUSION DIE SHAPE FOR 8" CONTAINER

The 300°F increase in the extrusion temperature therefore resulted in a 16% decrease in the extrusion constant over that experienced with the first 8" billet. Visual observation of the extrusion after sandblast revealed severe grain boundary tearings similar to that experienced in the previous low reduction ratio extrusion. The as-extruded sheet bar configuration and the surface tearing phenomena can be readily observed in Figure 33. Subsequent machining and ultrasonic evaluation showed that these ruptures extended almost to the center on 75% of the length leaving a nominal 12" long section satisfactory for rolling.

Two additional extrusions were made in order to evaluate further the scale-up to 8" diameter conditioned ingot. One extrusion was made through a rectangular die and the second extrusion was made using the tapered die previously described. Temperature was decreased from 3500° to 3200°F in an attempt to duplicate the results achieved in extruding 6" diameter ingot. The surface of the resultant extrusion indicated that surface tearing was still persisting but the results were considerably better than the previous attempts. Two additional billets were extruded at 3100°F, but the same surface problem resulted.

Three additional extrusion attempts were made in order to complete the extrusion effort for the final production phase. The extrusion parameters for the last three are shown in Table XXI. The first billet extruded to a 2.1" x 6" cross section did not clear the die, with the last 1/2" to 3/4" of the billet not extruded. The press did not stall since the recorded pressure was well below the stalling point. The problem was attributed to an inadequate length of carbon follower block to clear the extrusion from the die. The remaining two ingots were extruded at the same temperature and the results were satisfactory; however, the same surface tearing phenomena which were evident on previous attempts still persisted.



Picture 33

[illegible] Sheet [illegible]

TABLE XXI

EXTRUSION DATA FOR 8" DIAMETER CONDITIONED INGOT

Heat Number	Furnace Temperature	Transfer Time	Pressure (psi) Breakthrough	Pressure (psi) Running	Speed
KD1287	3000°F	32 Sec	90,000	82,000	9
KD1290	3000°F	33 Sec	90,000	82,000	9
KD1291	3000°F	31 Sec	90,000	78,000	9

Ultrasonic evaluation of the first extrusion which did not clear the die indicated it to be cracked except for a 15" length in the center. Due to hanging up in the die, this extrusion did not receive the standard slow cool which may have resulted in the cracking. It is also possible that the cracking occurred while manipulating it out of the press. The remaining two extrusions were sound except for normal nose and tail losses.

IV. Sheet Rolling Evaluation

A. Initial Rolling Studies

1. Sheet Bar Preparation and Application

In order to perform the initial investigation on sheet rolling variables, six sheet bars produced from the initial breakdown investigation were designated for rolling studies. Three directly extruded sheet bars shown in Figure 15 and three sheet bars press forged from 1-1/2" diameter round were sectioned to provide twelve starting pieces respectively. Prior to rolling, each piece was conditioned and inspected for internal and external defects. On the press forged sheet bar previously undetected cracks of a very small magnitude were discovered. Although visual observation indicated only a few cracks, dye penetrant inspection showed that they were frequent along both rounded edges of each piece and were transverse to the flat press forged edges. Apparently these cracks were heat checking resulting from the grinding operation. The depth of cracking was very shallow as repeated ultrasonic examination of the flat surface indicated completely sound material. An attempt was made to remove the cracks by additional grinding. However, this resulted in crack propagation. As there appeared to be no other satisfactory method of removing the cracks, rolling was initiated.

Figure 34 outlines the rolling parameters used. In order to hold the rolling variables to a minimum, cross rolling was not considered at this stage of the investigation. The processing was designed to provide three initial rolling temperatures, 2300°, 2500°, and 2700°F, two final rolling temperatures, and four different reductions from the last recrystallization anneal, all pieces finishing at a nominal .040" thick giving a total of 48 processing variables. Table XXII shows the application of each piece to the process outline.

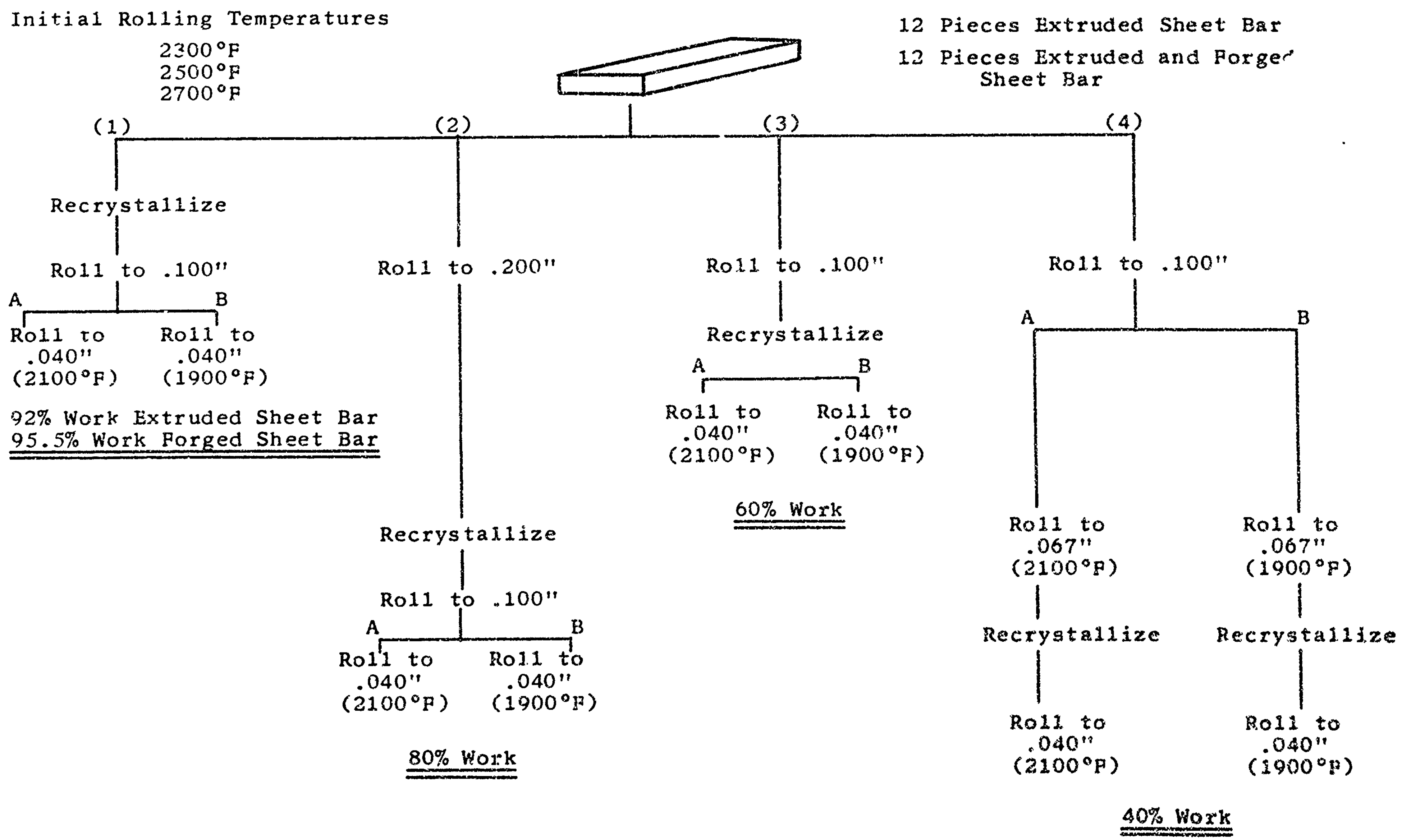


Figure 34

Preliminary Rolling Investigations

TABLE XXII

SUMMARY OF SHEET BAR APPLICATION

Identification	Series*	Sheet Bar Type	Starting Condition	Rolling Temperature (°F) Initial	Intermediate	Final	% Reduction From Recrystallization
Extrusion A							
A_1	(1)	Press Forged	Recrystallized	2300	--	2100** 1900	95.5
A_2	(2)	Press Forged	As-Forged	2300	2300	2100 1900	80
A_3	(3)	Press Forged	As-Forged	2300	--	2100 1900	60
A_4	(4)	Press Forged	As-Forged	2300	2300	2100 1900	40
Extrusion B							
B_1	(1)	Press Forged	Recrystallized	2500	--	2100 1900	95.5
B_2	(2)	Press Forged	As-Forged	2500	2300	2100 1900	80
B_3	(3)	Press Forged	As-Forged	2500	--	2100 1900	60
B_4	(4)	Press Forged	As-Forged	2500	2300	2100 1900	40
Extrusion C							
C_1	(1)	Press Forged	Recrystallized	2700	--	2100 1900	95.5
C_2	(2)	Press Forged	As-Forged	2700	2300	2100 1900	80
C_3	(3)	Press Forged	As-Forged	2700	--	2100 1900	60
C_4	(4)	Press Forged	As-Forged	2700	2300	2100 1900	40
Extrusion D							
D_1	(1)	Extruded	Recrystallized	2300	--	2100 1900	92
D_2	(2)	Extruded	Extruded	2300	2300	2100 1900	80
D_3	(3)	Extruded	Extruded	2300	--	2100 1900	60
D_4	(4)	Extruded	Extruded	2300	2300	2100 1900	40
Extrusion E							
E_1	(1)	Extruded	Recrystallized	2500	--	2100 1900	92
E_2	(2)	Extruded	Extruded	2500	2300	2100 1900	80
E_3	(3)	Extruded	Extruded	2500	--	2100 1900	60
E_4	(4)	Extruded	Extruded	2500	2300	2100 1900	40
Extrusion F							
F_1	(1)	Extruded	Recrystallized	2700	--	2100 1900	92
F_2	(2)	Extruded	Extruded	2700	2300	2100 1900	80
F_3	(3)	Extruded	Extruded	2700	--	2100 1900	60
F_4	(4)	Extruded	Extruded	2700	2300	2100 1900	40

* Refer to Figure 34

**Pieces Sectioned in Half For Final Rolling

In an effort to maintain consistency in the rolling parameters other than those variables being investigated, the following were held constant regardless of rolling temperature or total reduction desired.

1. Furnace air atmosphere.

2. Soaking time - 5 minutes at temperature.

3. Roll speed - 50 RPM (144 SFPM)

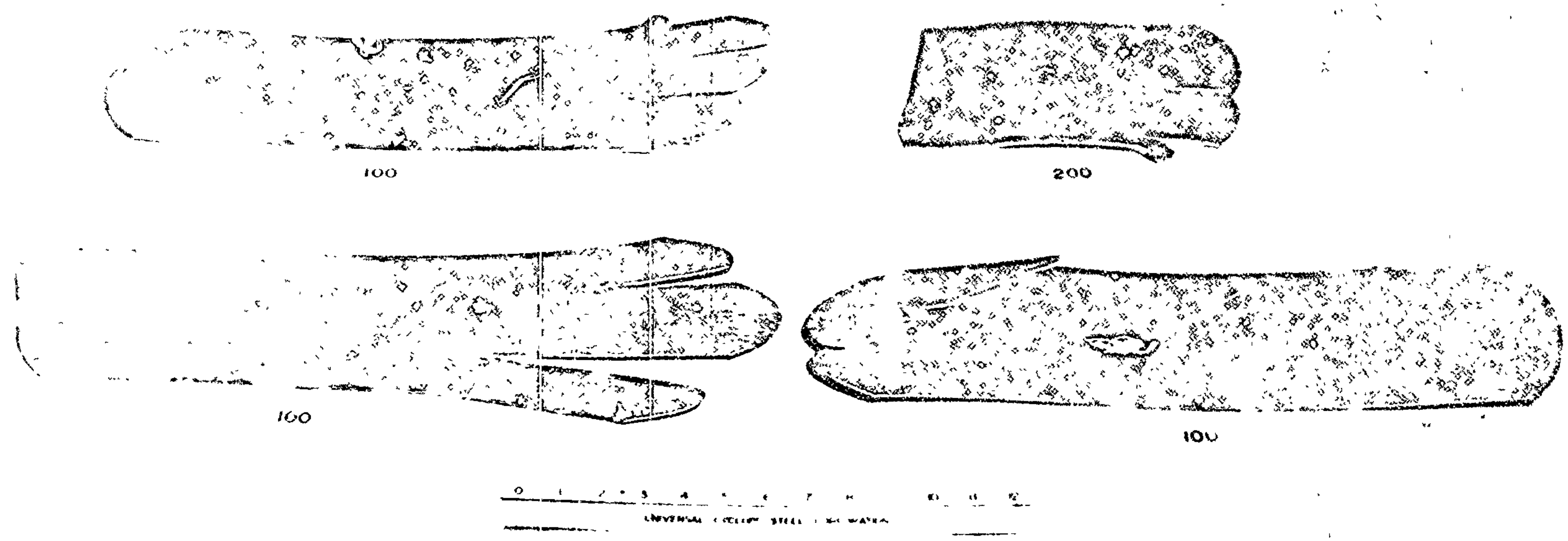
2. Rolling Characteristics

a. Initial Rolling

Observation of the initial rolling operation revealed no noticeable difference in the rolling characteristics due to temperature variation. The recrystallized sheet bars appeared to roll identical to the remaining as-extruded or as-forged sheet bar. The major problem incurred during the rolling operation was crack propagation on the press forged sheet bars, in which light edge cracking had been detected prior to rolling. Figure 35 shows the results of initial rolling of the press forged sheet bars and is typical of the results obtained with press forged sheet bars. In addition to the extensive longitudinal cracks, many shorter cracks are present on the ends. These cracks were the result of the press forging operation which has been discussed previously in this report.

Of the twelve extruded sheet bar pieces, one had a detectable crack prior to rolling. The effect of starting with completely sound material is shown in Figure 36 for Extrusion E and is typical for extruded sheet bar. Every piece, except the one in which the crack was detected, rolled to completely sound sheet.

Visual observation of all sheets in the as-rolled condition indicated very good surface. After sandblasting,



INITIAL ROLLING OF SHEET BAR B

Figure 35

Intermediate Sheet from Extrusion B
Rolling Temperature 2500°F - Press Forged Sheet Bar

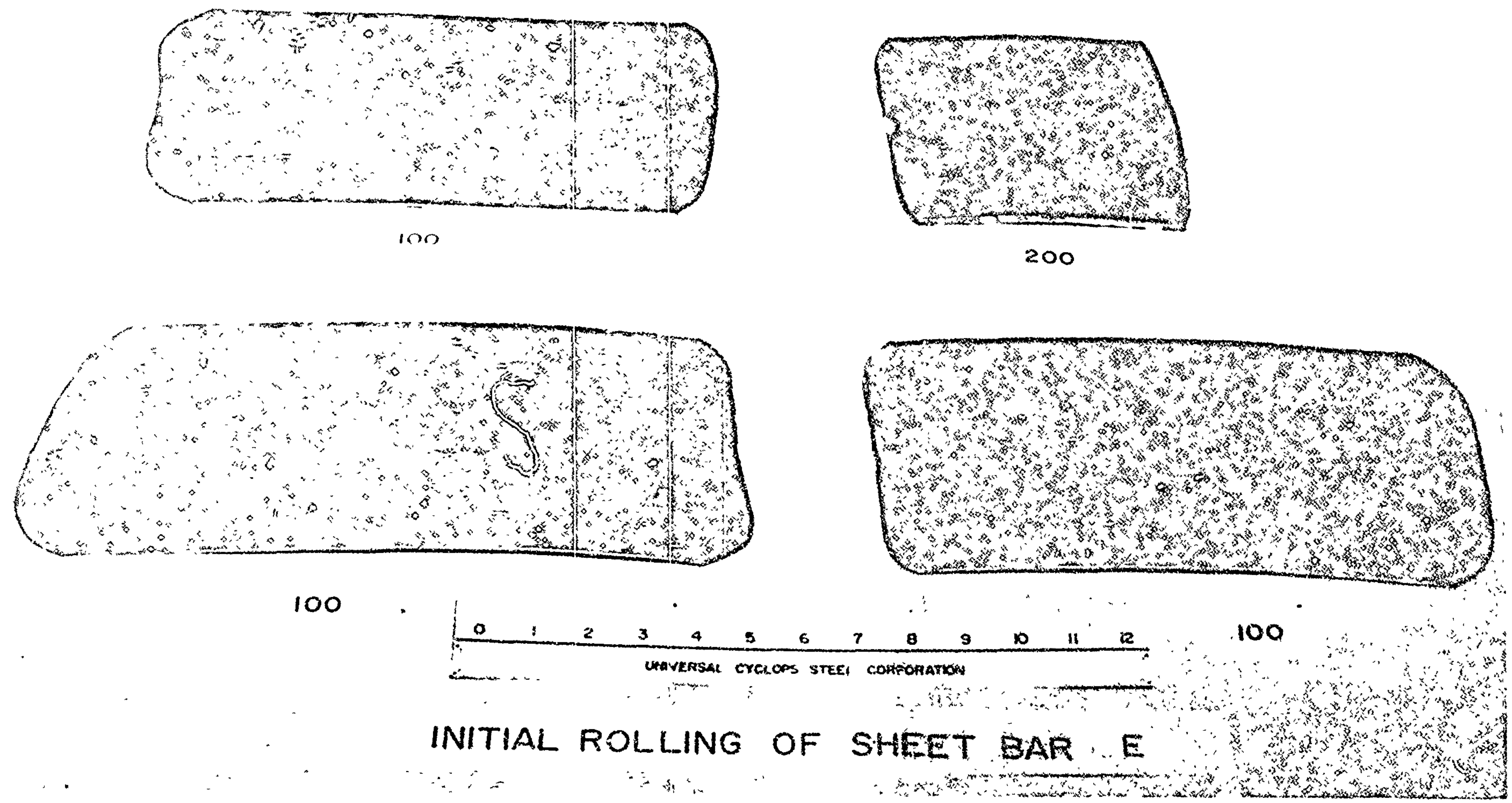


Figure 36

Intermediate Sheet From Extrusion E
Rolling Temperature 2500°F - Extruded Sheet Bar

which removed the oxide scale, an erosion or corrosion effect was observed to some extent on all pieces. The severity of the corrosion effect appears to correlate with the rolling temperature. For example, the pieces rolled at higher temperatures were more severely eroded. This effect can be attributed to localized liquid oxide formation prior to removal from the furnace. At this point in the processing schedule, all pieces were conditioned by abrasive cutting all cracked edges and sandblasting for further rolling.

The No. 2 and No. 3 series as described in Table XXII were recrystallized at .200" gauge and .100" gauge respectively to permit the desired degree of hot-cold work at the finished gauge of .040". In order to determine the recrystallization temperature at this intermediate gauge, samples were heat treated for one hour over a temperature range of 2000° to 2600°F at 100°F increments. Figures 37 through 40 are plots of the hardness after each respective annealing treatment. In all cases, the curves show the effect of rolling temperature on the resulting hardness drop due to heat treatment. Note that the as-rolled hardness decreases with increasing rolling temperature. The estimated recrystallization as determined by microstructural observation is also shown on the hardness-stress relief curves.

The as-rolled microstructures for material receiving constant reductions with variable rolling temperatures are shown in Figure 41. The grain size increases as the rolling temperature increases. Figure 41 shows that in-process recrystallization occurs when rolling at 2700°F. The increased reduction of the material rolled 87% resulted in an even larger grain size differential than the material given 73% reduction.

One-hour anneals at various temperatures show little effect in retaining the cold-worked structure due to

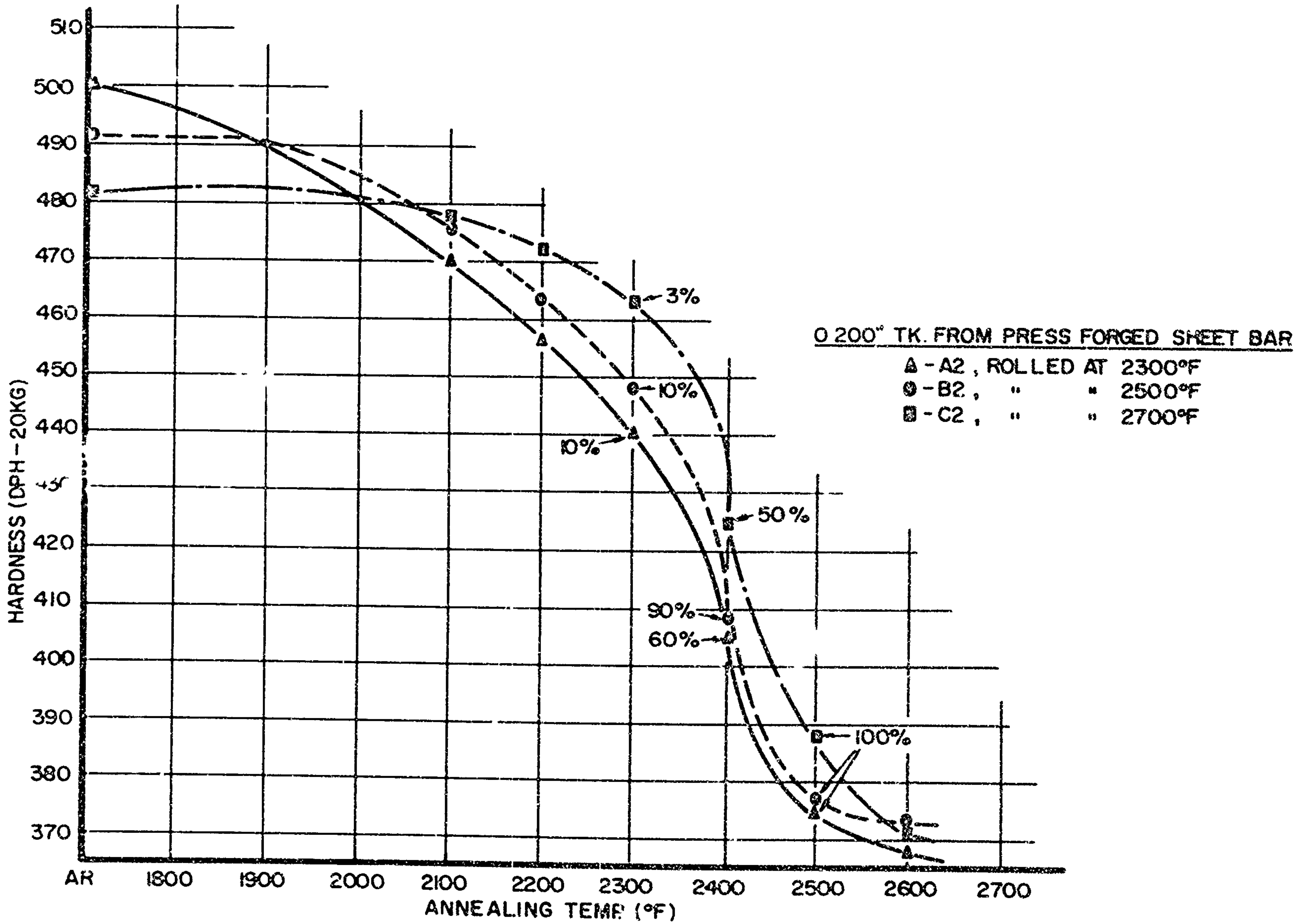


FIGURE 37

EFFECT OF INITIAL ROLLING TEMPERATURE ON
RESPONSE TO HEAT TREATMENT (REDUCTION 73.5%)

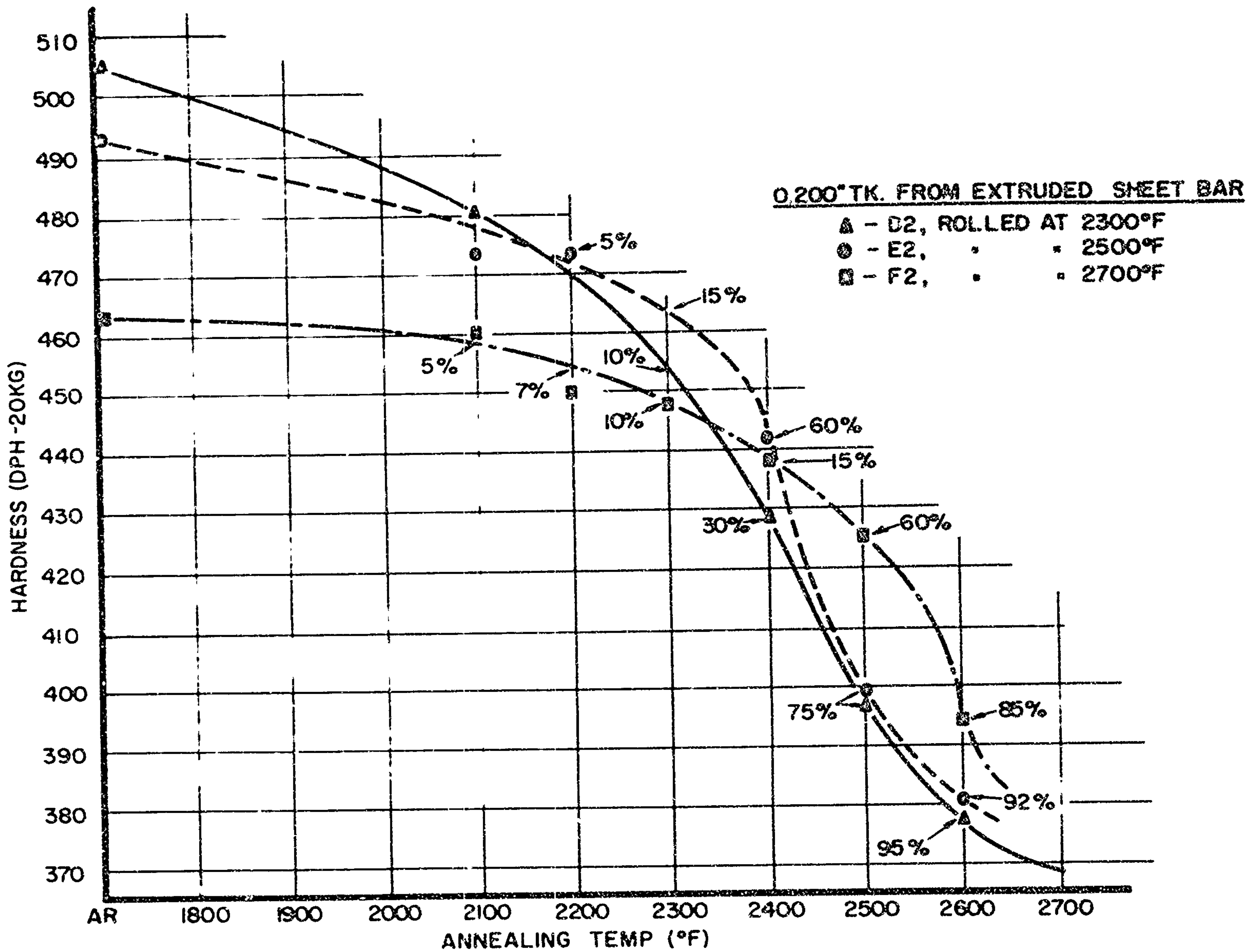


FIGURE 38

EFFECT OF INITIAL ROLLING TEMPERATURE ON
RESPONSE TO HEAT TREATMENT (REDUCTION 73.5%)

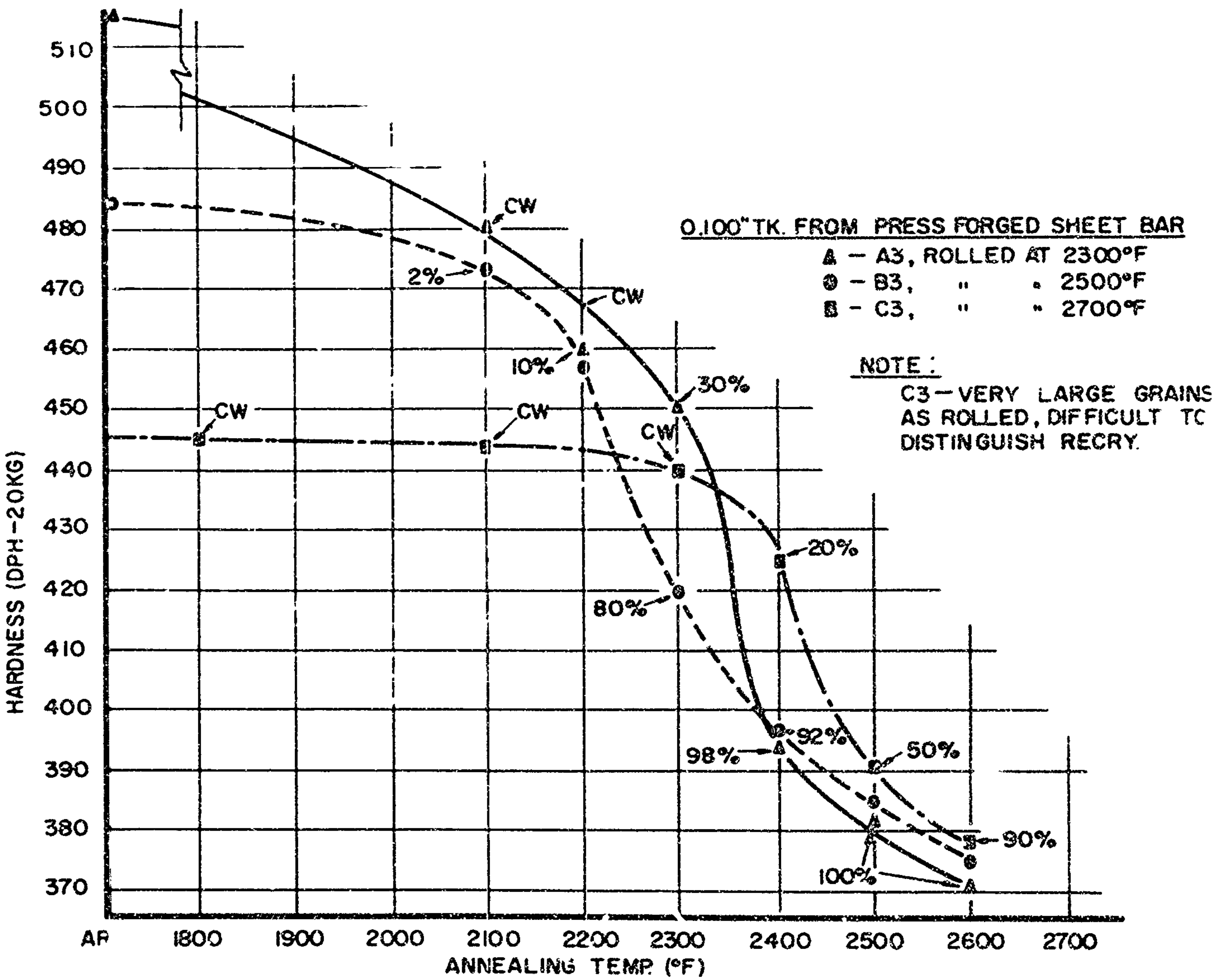


FIGURE 39

EFFECT OF INITIAL ROLLING TEMPERATURE ON RESPONSE TO HEAT TREATMENT (REDUCTION 87%)

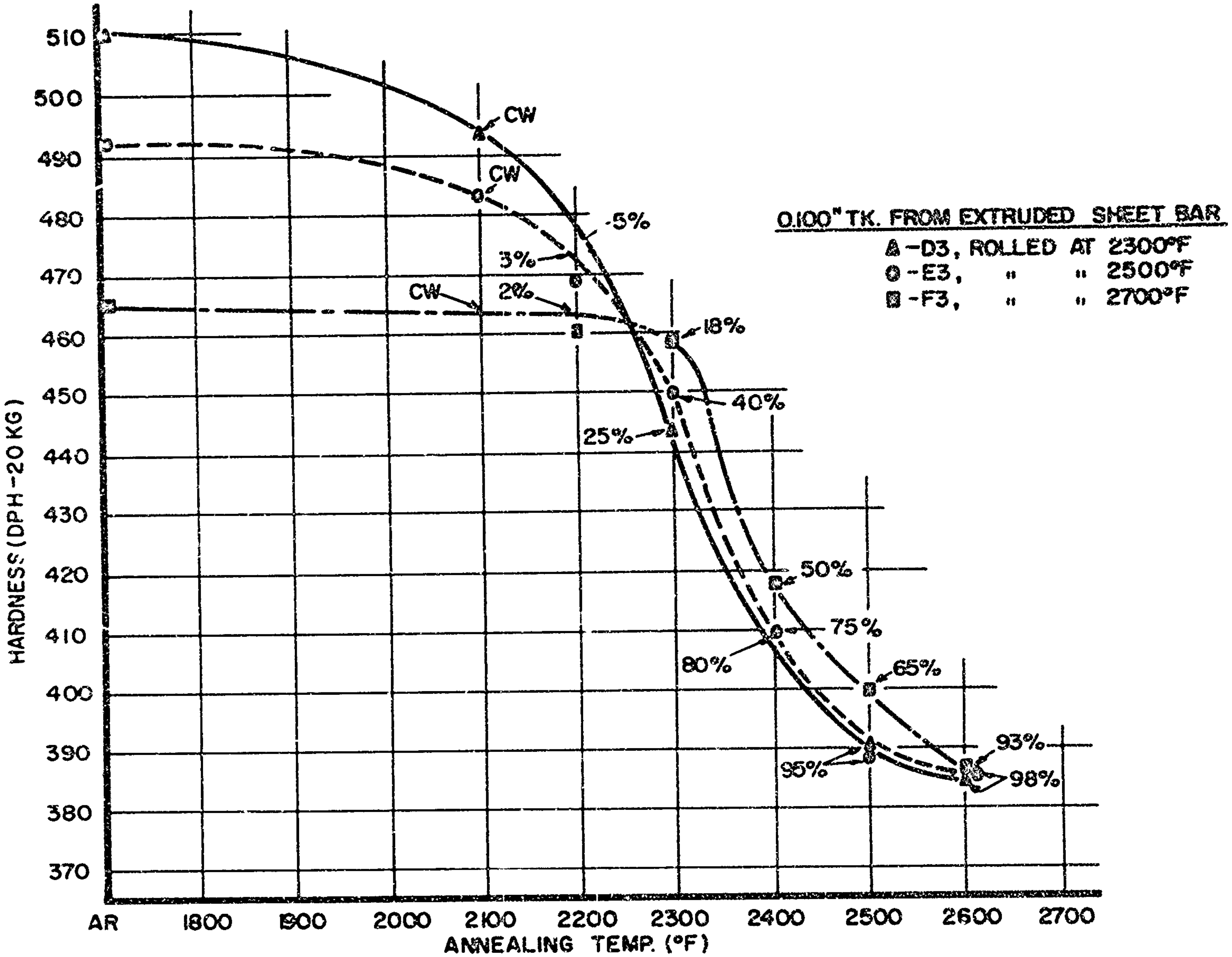
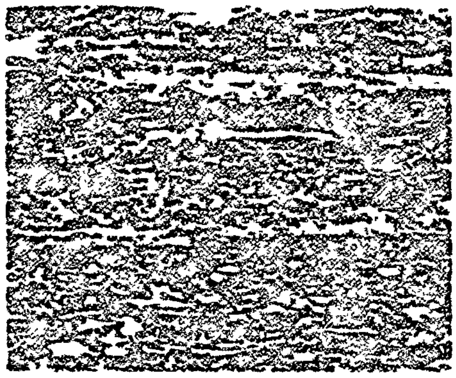


FIGURE 40

EFFECT OF INITIAL ROLLING TEMPERATURE ON RESPONSE TO HEAT TREATMENT (REDUCTION 87%)

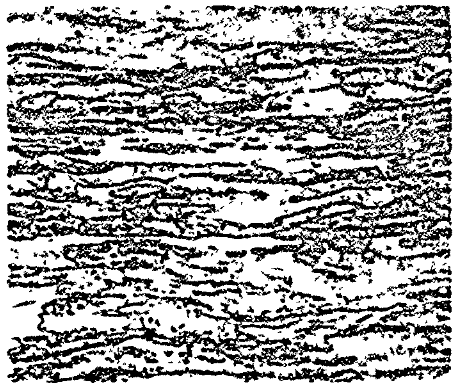
R10680 D_2



R10671 A_3

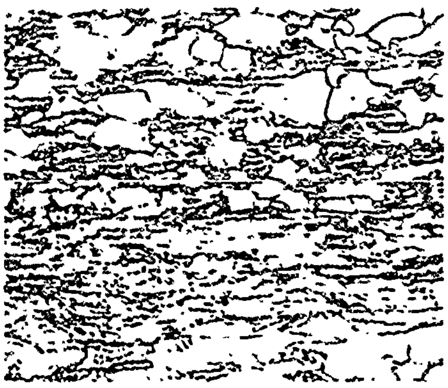
2300°F Rolling Temperature

R10681 E_2



R10672 B_3

2500°F Rolling Temperature



R10682 E_2

R10674 C_3

2700°F Rolling Temperature

Constant Reduction - 73.5%
Constant Gauge - 0.200"

Extruded Sheet Bar

Constant Reduction - 87%
Constant Gauge - 0.100"

Press Forged Sheet Bar

Figure 41

As-Rolled Microstructure for Variable
Starting Materials and Rolling Temperatures

higher rolling temperatures. The high and low temperature rolled material indicated an equivalent degree of recrystallization after annealing at 2600°F.

b. Intermediate Rolling

For the second rolling operation, series 1 was rolled in the as-rolled condition from 0.100" to 0.040", series 2 in the recrystallized condition from 0.200" to 0.100", series 3 in the recrystallized condition from 0.100" to 0.040", and series 4 in the as-rolled condition from 0.100" to 0.067". Inadvertently, five of the twelve pieces in series 4 were rolled directly to the final 0.040" gauge without being recrystallized at 0.067". Although this eliminated some of the rolling variables being studied, it provided material for evaluation containing 99+% reduction, i.e. this material received no intermediate anneal from ingot to final sheet.

At this stage, samples from series 4 were heat treated to determine recrystallization temperatures. Figures 42 and 43 show plots of hardness versus annealing temperature along with estimated percent recrystallization for these samples. The identification of the samples has picked up a third digit, i.e. F_4A, F_4B indicating, as shown in Figure 34, that the sheet was sectioned into two pieces for final rolling. The A_4A, B_4B, C_4A, and E_4A sheets not plotted are those which were rolled directly to 0.040".

These curves, like those from initial rolling, show that the higher rolling temperatures result in lower as-rolled hardness. However, on this series, the intermediate rolling was accomplished at a constant 2300°F and the difference in as-rolled hardness due to the initial rolling temperature variations was not as great.

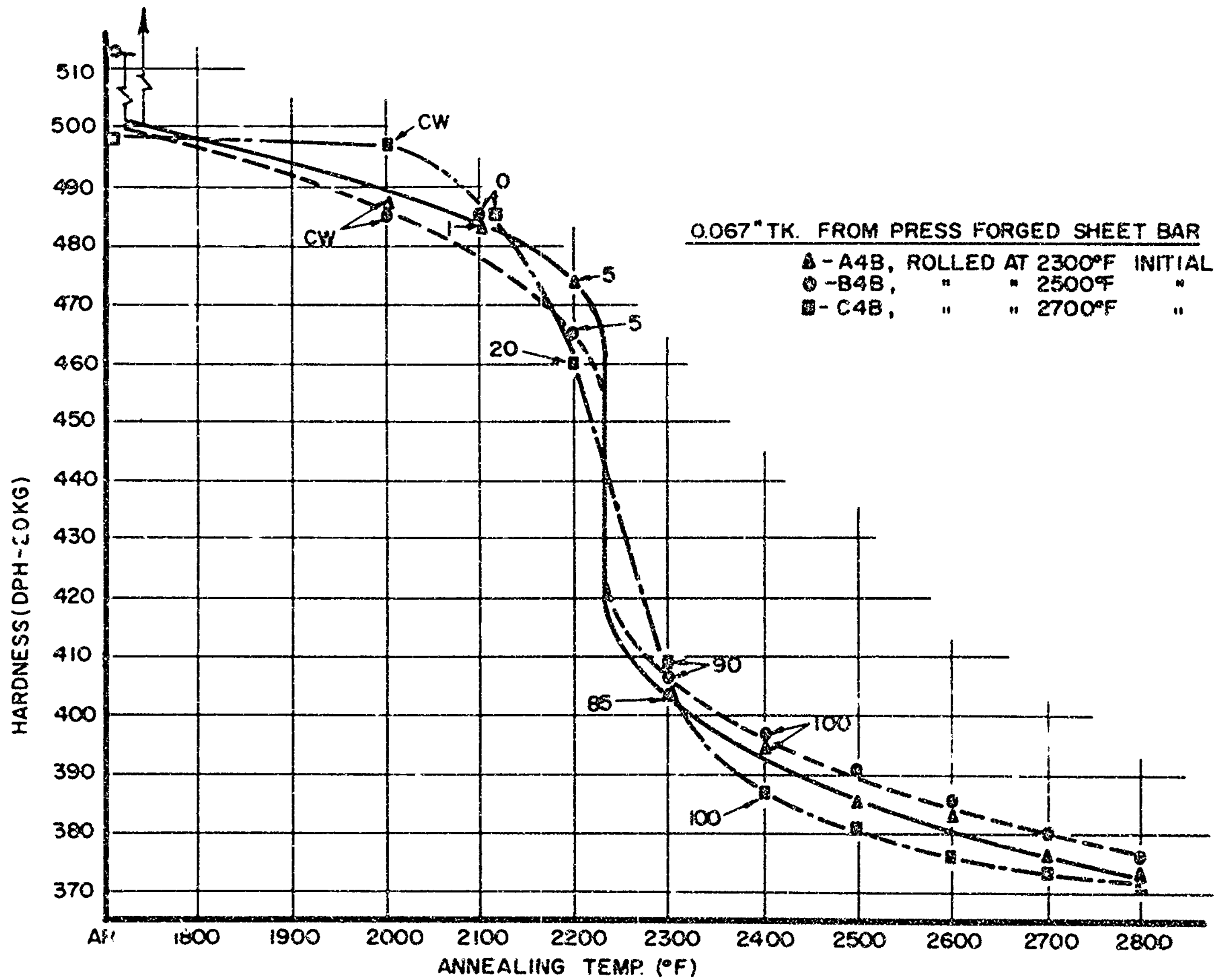


FIGURE 42

EFFECT OF INITIAL ROLLING TEMPERATURE AFTER INTERMEDIATE ROLLING AT 2300°F ON RESPONSE TO HEAT TREATMENT (REDUCTION 91.5%)

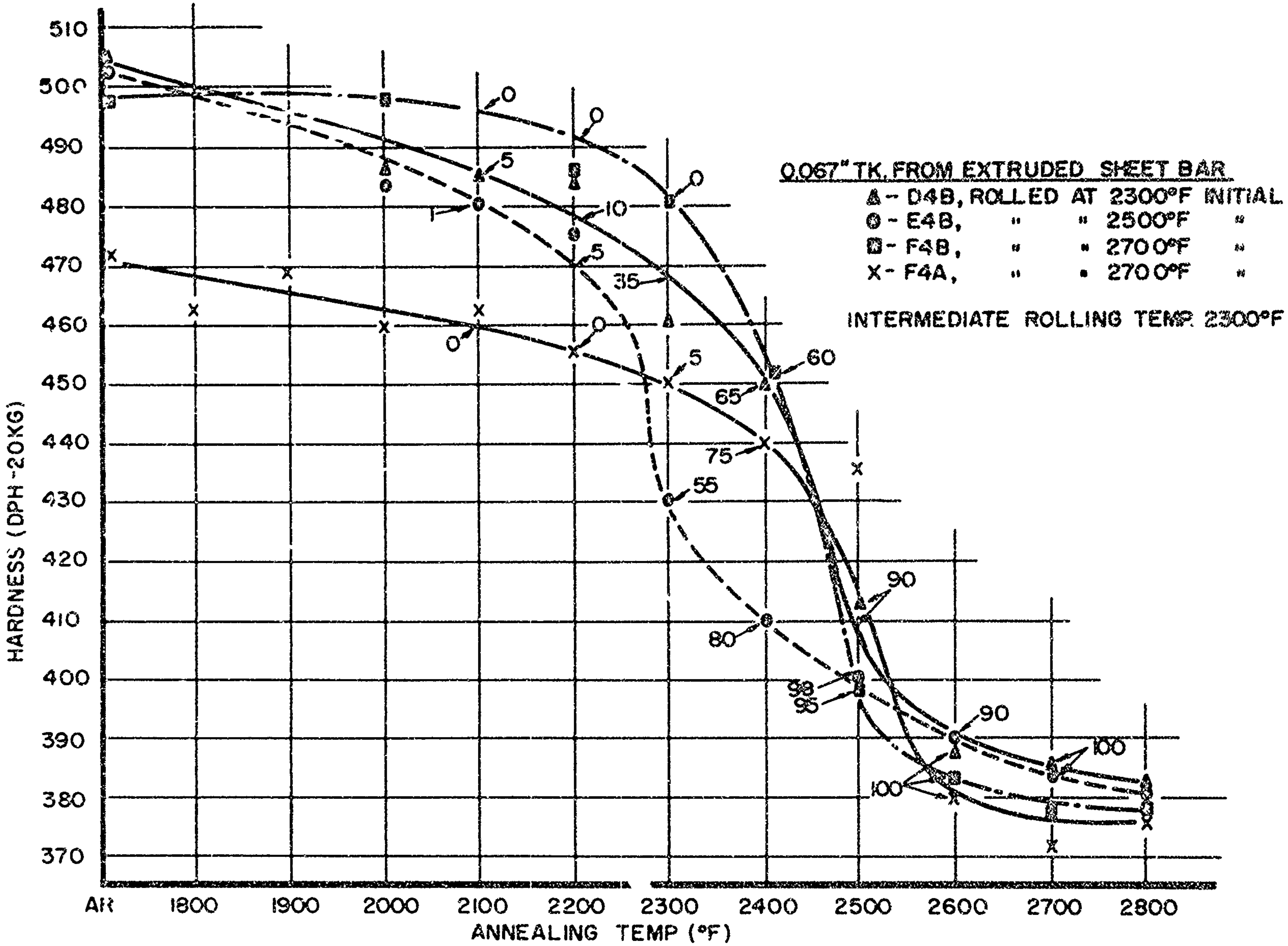


FIGURE 43

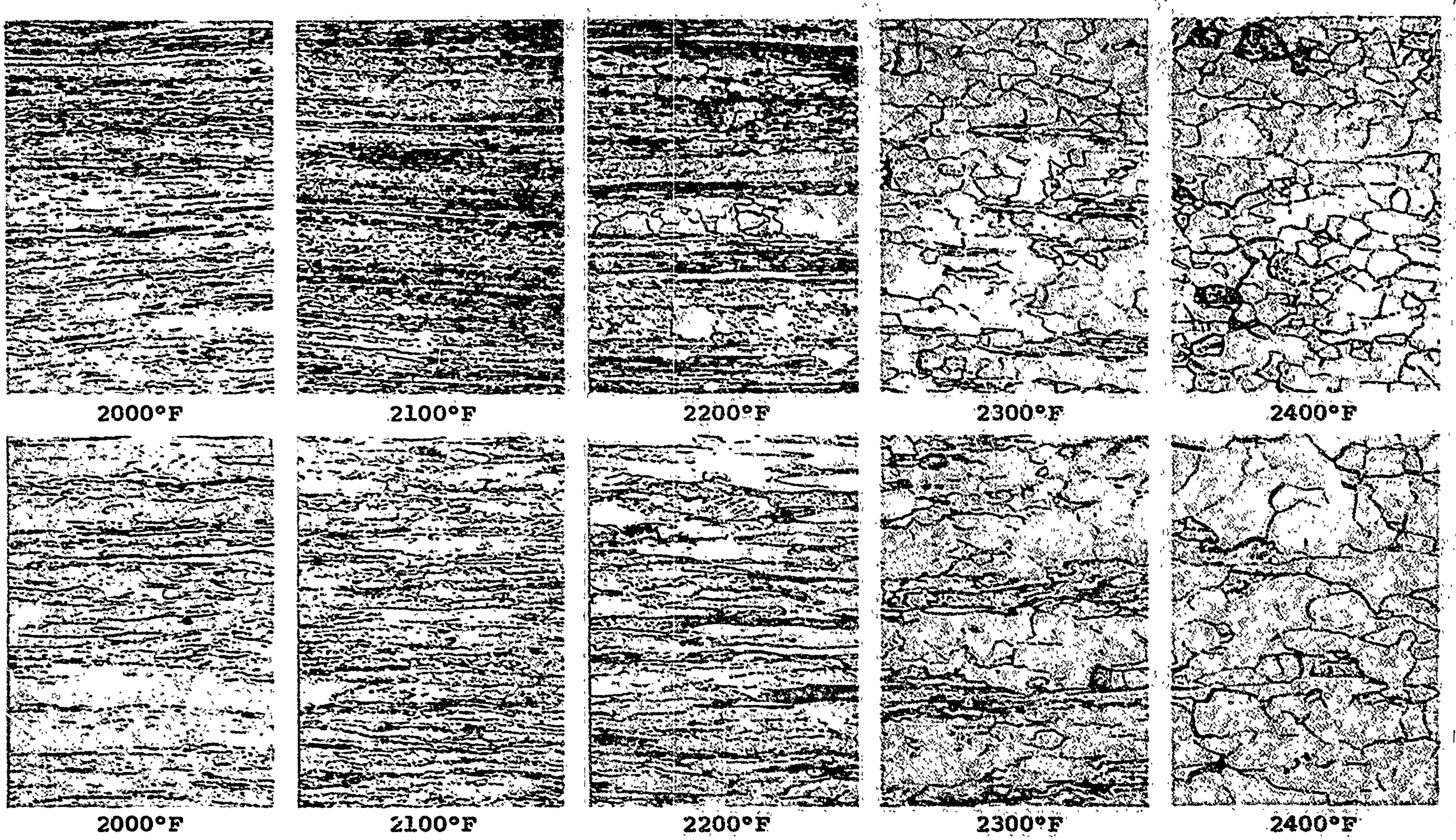
EFFECT OF INITIAL ROLLING TEMPERATURE AFTER INTERMEDIATE ROLLING AT 2300°F ON RESPONSE TO HEAT TREATMENT (REDUCTION 91.5%)

F_4A, as shown in Figure 43, has an appreciably lower hardness than the average. This can be attributed to the fact that it was the first piece rolled to 0.067" and required a reheat prior to the last pass. All other pieces in this series were rolled from 0.100" to 0.067" without reheating. The microstructure at the 0.067" gauge showed more severe work than that previously shown for 0.100" gauge material. Figure 44 shows the effect of various annealing treatments on material initially rolled at 2300° and 2700°F. The material rolled at 2300°F had begun to recrystallize at 2200°F while the 2700°F rolled material still retained its cold-work structure. At 2400°F, however, both show an equal degree of recrystallization with the 2700°F rolled material having a much larger recrystallized grain size.

The previous curves on series 2, 3, and 4 have shown the effect of rolling temperatures on the as-rolled and heat treated hardness. Rolling to this point has also resulted in various reductions from sheet bar. Figure 45 shows the effect of these reductions on the hardness after stress relief. As expected, increased percent reduction results in increased hardness and an accelerated recrystallization rate. However, the 100% recrystallization temperature is relatively constant for the three different reductions.

c. Final Rolling

In the third rolling operation, the series 2 material was rolled in the as-rolled condition from 0.100" to 0.040" and the series 4 material in the recrystallized form from 0.067" to 0.040". For all final rolling, including the series 1 and 3 material rolled in the second operation, stainless steel cover plates were utilized. These plates were necessary for the following reasons:



Top Series — A_4B Initial Rolling Temperature - 2300°F — One Hour Annealing Treatment
Bottom Series — C_4B Initial Rolling Temperature - 2700°F — Magnification - 200X

Figure 44

Microstructures of Progressive Annealing Treatments
Low Versus High Temperature Rolling

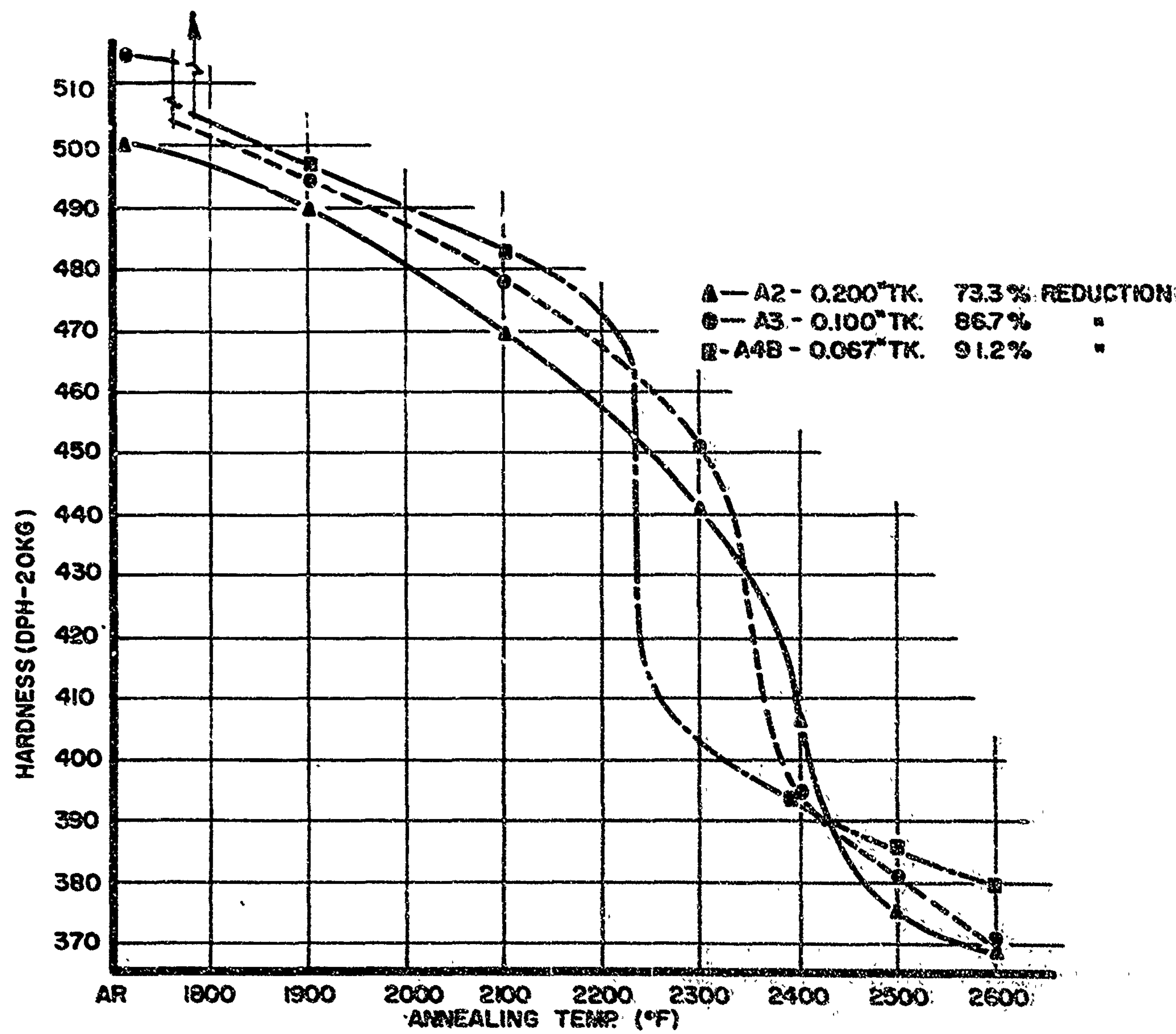


FIGURE 45

EFFECT OF % REDUCTION ON RESPONSE TO HEAT TREATMENT
(CONSTANT ROLLING TEMPERATURE 2300°F)

1. The pilot mill used did not have a sufficient separating force to permit single sheet reductions below 0.090" without frequent reheats. The cover plates thus permitted greater reductions in producing the 0.040" gauge.

2. At the thinner gauges, the high thermal conductivity of tungsten results in a rapid heat loss once the material is removed from the furnace. The cover plates assisted in maintaining heat in the sheet.

3. The cover plates prevent pickup of contamination from the furnace hearth and also minimize oxidation.

Rolling at 2000°F and above with stainless steel cover plates resulted in a bonding between the tungsten and stainless steel, especially if the contact surfaces were clean. In order to prevent this bonding, the tungsten sheets were heated to a temperature which would permit the formation of an oxide coating when removed from the furnace. The sheet was then placed between two cover plates, heated and rolled. The oxide film prevented any bonding from occurring.

3. Evaluation of Rolled Sheet

a. Surface Finish

In evaluating the surface finish, the oxide film must be removed. In the as-rolled condition, the sheets were extremely brittle and could not be sandblasted. An alternate method of removing the oxide while retaining the as-rolled surface was hot caustic cleaning. This also did not appear satisfactory because

of the severe water quench after removal from the 900°F caustic. However, by heat treating in hydrogen, the oxide was reduced, leaving a loose film which could be wiped off. The general appearance of the surface could best be described as a matte finish. Lower rolling temperatures, when utilizing cover plates, should improve this surface since the cover plates deformed too readily at the temperatures utilized. Rolling without cover plates on a four-high mill might also be desirable.

b. Cracking

One piece cracked during intermediate rolling at .200" thick. This resulted after attempting to take two passes per reheat following a recrystallization anneal. This tended to confirm previous experience on the criticality of deformation after recrystallization. The only cracking in the final rolling operations resulted when the edge of a piece slipped out of the cover plates prior to or during rolling. Cracks then resulted from non-uniform deformation. Two sheets were cracked in removing them from the cover plates because of bonding. Two additional sheets were cracked subsequent to the rolling operation which was attributed to mishandling. It appeared from this work as well as several corporate sponsored programs, that handling of material is one of the major problems associated with tungsten production. This is especially true when the material is in the form of sheet.

c. Lamination Tendency

"Lamination" is defined as a defect, in the form of a physical separation or layering within a sheet parallel to the plane of the sheet. This defect may be infinitely small but can be detected by microscopic or ultrasonic inspection. The term "delamination" is used to describe a physical separation, not present after rolling, but which occurs during shearing or forming operations.

In shearing samples for hardness and microscopic evaluation, delaminations were visible on the polished surface of many sheets. At this point, it was not known whether these laminations or delaminations were inherent to the rolling operation or resulted from shearing. Additional samples were sheared and then abrasive cut transverse to the sheared edge. They were subsequently polished and examined along the abrasive cut edge. Separations existed on each specimen observed, with the separation initiating at the sheared edge and extending from 0.100" to 0.200" in depth. Beyond this, the material was sound, suggesting that the separation resulted from the shearing operation rather than rolling and thus could be classified as delaminations. Laminations were detected along the extreme edges and ends of several sheets, however, no correlation could be made with the rolling practice. These laminations undoubtedly are due to non-uniform work of the edges and ends of the sheet. From this investigation, it was apparent that the shearing operation was highly critical and improved techniques would be necessary.

d. Stress Relief and Recrystallization Characteristics

Samples were cut from each sheet and annealed for one hour over the range of 1800° to 2600°F at 100°F increments. Appendix IV contains a summary of the annealing curves plotted for all material at the final .040" gauge. This series of curves shows the effect of annealing on the hardness for identical starting material rolled to different reductions. A summary of these curves is shown in Table XXIII where the average of four samples rolled under similar conditions, i.e. A_1A, A_1B, D_1A, D_1B all initially rolled at 2300°F to the same gauge, are listed for each rolling temperature and stress relief condition. These averages show that the initial rolling temperature has little effect on the resulting hardness in

TABLE XXIII

AVERAGE HARDNESS FOR .040" SHEET BY ROLLING TEMPERATURE AND REDUCTION

Reduction	As Rolled	Annealing Temperature (°F) 1800	1900	2000	2100	2200	2300	2400	2500	2600
2300°F										
92-95	509	502	492	482	469	445	409	389	381	376
80	503	499	497	489	470	461	412	390	380	376
60	494	485	482	477	472	458	431	398	378	375
40	467	467	467	466	465	459	434	406	389	373
Average	493	488	485	479	469	456	422	396	382	375
2500°F										
92-95	504	492	490	485	471	440	401	385	379	377
80	499	495	493	487	477	444	412	386	378	374
60	488	476	476	474	471	463	429	391	376	372
40	468	468	468	466	465	458	425	390	374	368
Average	490	483	482	478	471	451	417	388	377	373
2700°F										
92-95	505	502	499	489	478	452	410	387	379	374
80	494	498	495	490	480	456	410	360	375	371
60	491	486	485	485	476	462	424	397	381	373
40	472	472	472	471	469	462	448	411	382	370
Average	491	488	488	484	476	458	423	389	379	372
All Samples										
92-95	506	499	494	485	473	446	407	387	380	376
80	499	495	495	489	476	454	411	379	378	374
60	491	482	481	479	473	461	428	395	378	373
40	469	469	469	468	466	460	436	402	382	370
Average	491	486	485	480	472	458	421	391	380	373

the as-rolled condition or after heat treatment. Conversely, the curves show that the as-rolled hardness and recrystallization rate increase with increasing reductions.

e. Metallographic Evaluation

A review was made on all .040" material in the as-rolled and annealed condition. In general, it can be stated that the large grain size resulting from initial high temperature rolling is retained in the final structure. The annealing temperature required to initiate recrystallization increased with decreasing reductions. Table XXIV shows the annealing temperature required to produce specific degrees of recrystallization in comparison with the percent reduction.

TABLE XXIV

RECRYSTALLIZATION VERSUS REDUCTION .040" SHEET

	One Hour Annealing Temperature (°F) to Produce Indicated Percent Recrystallization				
Reduction	Initiation	10-20%	50-60%	80-90%	98-100%
99+%	1900	2100	2150	2200	2400
92-95%	1900	2100	2150	2200	2400
80	2100	2200	2300	2350	2450
60	2200	2300	2350	2500	2600
40	2300	2400	2500	2550	2650

The effect of rolling reductions and temperatures on the microstructure is shown in Figure 46. The rolling temperatures shown are those for initial breakdown, the finish rolling temperature being either 2100° or 1900°F for all sheets, which, in itself, did not affect the visible structure. It can be seen that increasing reductions and decreasing temperatures have a significant effect on refining the wrought grain structure.

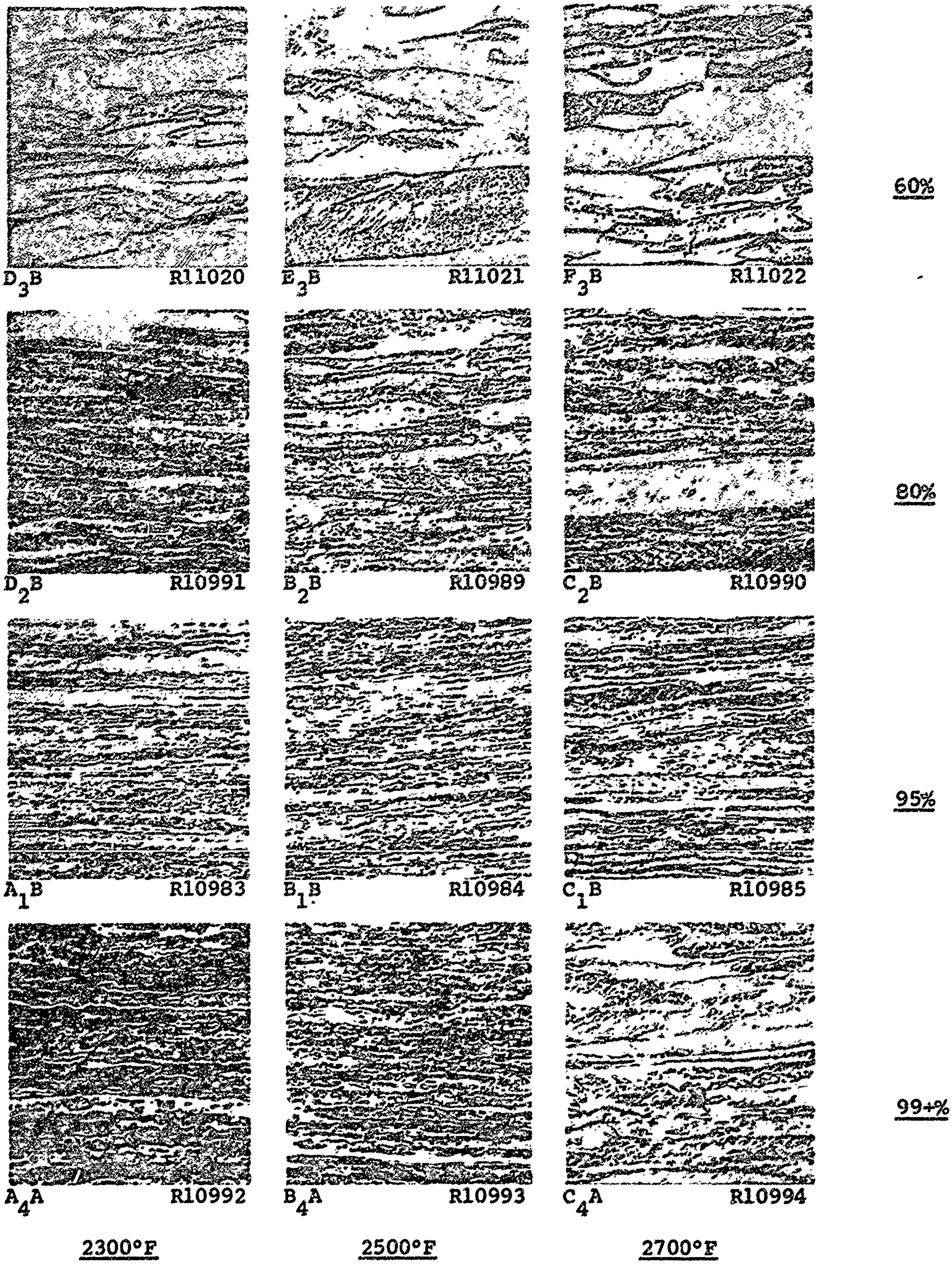


Figure 46

As-Rolled Microstructures - Rolling Temperature Vs Reduction
Magnification - 200X

f. Bend Transition

Bend tests were run on all material rolled. Testing was accomplished on a Tinius Olsen tensile machine at the maximum available bend rate of 8" per minute. Four (4) T bends were made utilizing the simple beam support method with a resistance muffle-type furnace. Specimens were annealed for one hour at 100°F increments from 1800° to 2200°F. Subsequent preparation consisted of pickling in 60% HNO_3-40% HF (by volume) and polishing the edges on 150 grit paper.

The transition temperature was not found on all sheets because of the limitation of specimens for each condition. Less than (<) symbols are used to designate material which bent successfully at the lowest temperature checked. Conversely, greater than (>) symbols indicate that the material broke at the highest temperature checked.

The longitudinal transition temperature obtained are summarized in Table XXV. Only those samples having 92% or greater reduction demonstrated a transition temperature of 275°F or lower. The sheets initially rolled at 2700°F are shown to have a higher transition temperature than the same material rolled at 2300° and 2500°F. Review of grain structure proved conclusively that increased wrought grain size resulted in increased transition temperatures. The lowest longitudinal transition temperature was 200°F. Three additional sheets had a transition temperature of 225°F.

Bend transition in the transverse direction was limited to material having 92% or greater reduction, as this material tested in the longitudinal direction was shown to be superior. The results of the transverse tests are shown in Table XXVI. The higher temperature stress relief anneals resulted in increased

TABLE XXV

LONGITUDINAL BEND TRANSITION TEMPERATURES

% Reduction	Identification	Rolling Temperature (°F) Initial	Intermediate	Final	Bend Transition After Indicated Annealing 1800°F	1900°F	2000°F	2100°F	2200°F	1700°F
99+	A_4A	2300	2300	2100	225	275	425	350	375	325
	B_4A	2500	2300	2100	275	275	350	<325	275	275
	C_4A	2700	2300	2100	<525	<375	>425	>400	>425	--
	D_4A	2300	2300	2100	275	275	425	400	425	275
	E_4A	2500	2300	2100	275	275	>425	400	375	275
95	A_1A	2300	--	2100	375	325	350	--	475	325
	A_1B	2300	--	1900	275	275	275	350	475	275
	B_1A	2500	--	2100	350	325	375	375	475	300
	B_1B	2500	--	1900	300	275	350	375	>475	300
	C_1A	2700	--	2100	>475	>475	>475	>475	--	--
	C_1B	2700	--	1900	400	400	425	425	>475	--
92	D_1A	2300	--	2100	225	275	350	350	375	200
	D_1B	2300	--	1900	225	275	375	400	400	325
	E_1A	2500	--	2100	400	275	375	425	425	275
	E_1B	2500	--	1900	325	425	400	--	>475	275
	F_1A	2700	--	2100	400	325	400	475	475	
	F_1B	2700	--	1900	275	350	>475	425	475	
80	B_2A	2500	2300	2100	375	425	425	450	>475	
	B_2B	2500	2300	1900	425	425	450	<475	525	
	C_2A	2700	2300	2100	325	>475	525	475	>475	
	C_2B	2700	2300	1900	475	425	>475	--	525	
	D_2A	2300	2300	2100	325	<375	<375	>475	525	
	D_2B	2300	2300	1900	<425	<375	425	<475	525	
	E_2A	2500	2300	2100	<475	375	425	<475	>525	
	E_2B	2500	2300	1900	425	450	>525	--	>525	
	F_2A	2700	--	2100	475	425	<375	--	475	
	F_2B	2700	--	1900	400	<375	<375	--	>525	
60	A_3A	2300	--	2100	>475	525	>525	<525	525	
	A_3B	2300	--	1900	<475	525	>525	--	<525	
	B_3A	2500	--	2100	525	525	525	<525	<525	
	B_3B	2500	--	1900	525	525	<475	<525	<525	
	C_3A	2700	--	2100	>525	>525	>525	>525	>525	
	C_3B	2700	--	1900	>525	>525	>525	>525	>525	
	D_3A	2300	--	2100	>525	<475	>525	>525	>525	
	D_3B	2300	--	1900	>525	525	>525	>525	>525	
	E_3A	2500	--	2100	475	>525	>525	>525	>525	
	E_3B	2500	--	1900	>525	>525	525	>525	>525	
	F_3A	2700	--	2100	>525	<475	>525	>525	>525	
	F_3B	2700	--	1900	525	<475	525	>525	>525	
40	F_4A	2700	2300	2100	>525	>525	>525	>525	>525	
	A_4B	2300	2300	1900	>525	>525	>525	>525	>525	
	B_4B	2500	2300	1900	>525	>525	>525	>525	>525	
	C_4B	2700	2300	1900	>525	>525	>525	>525	>525	
	D_4B	2300	2300	1900	>525	>525	>525	>525	>525	
	E_4B	2500	2300	1900	>525	>525	>525	>525	>525	
	F_4B	2700	2300	1900	>525	>525	>525	>525	>525	

TABLE XXVI

TRANSVERSE BEND TRANSITION TEMPERATURES

% Reduction	Identification	Rolling Temperature (°F)		Bend Transition After Indicated Annealing		
		Initial	Final	1800°F	1900°F	2000°F
99+	A_4A	2300	2100	425	450	475
	B_4A	2500	2100	450	500	400
	D_4A	2300	2100	400	400	450
	E_4A	2500	2100	450	475	475
95	A_1A	2300	2100	400	475	475
	A_1B	2300	1900	375	475	425
	B_1A	2500	2100	340	425	475
	B_1B	2500	1900	425	475	500
92	D_1A	2300	2100	400	450	475
	D_1B	2300	1900	375	450	475
	E_1A	2500	2100	400	400	475
	E_1B	2500	1900	400	425	475

transition temperatures which agree with the analysis of longitudinal testing. Although the transition temperature range was not great for the specimens annealed at 1800°F, the sheet with the lowest temperature, 350°F, does not correspond to the lowest longitudinal transition. However, when both the longitudinal and transverse data are compared, four sheets show the most desirable combination. The processing schedules and resulting transition temperatures of these four sheets are shown in Table XXVII.

In reviewing all of the bend transition data, the following observations were made:

1. Bend transition results indicated a slight advantage with the extruded sheet bar over the press forged sheet bar as a starting material.

2. Material rolled at 2500° and 2700°F in the initial rolling operation showed higher bend transition due to larger grain size resulting from in-process recrystallization.

3. The optimum bend transition properties were obtained from material containing a minimum of 92% reduction from the last recrystallization anneal.

4. Stress relief to improve bend transition must be accomplished at temperatures below the temperature of initial recrystallization.

g. <u>Tensile Testing</u>

Tensile data was established for all material rolled. Testing procedures utilized were as follows:

TABLE XXVII

COMPARISON OF LONGITUDINAL AND TRANSVERSE
BEND TRANSITION FOR FOUR SELECTED SHEETS

Sheet Bar	% Reduction	Rolling Temperature		Stress Relief	Transition Temperature	
		Initial	Final		Longitudinal	Transverse
Press Forged	95	2300°F	2100°F	1800°F	375*	400
Press Forged	95	2300°F	1900°F	1800°F	275	375
Extruded	92	2300°F	2100°F	1800°F	225	400
Extruded	92	2300°F	1900°F	1800°F	225	375

*This transition temperature is high; however, it is inconsistent with other stress relief data and considered erroneous. Non-availability of additional material prevented rechecking this point.

Specimen (See Figure 47)

0.75" Gauge Length
0.187" Gauge Width

Test Temperature - 900°F

Strain Rate - 0.005"/in/min to .6% Yield
0.050"/in/min to Fracture

Table XXVIII lists the tensile data established for the five stress relief conditions utilized. Note that a progressively greater number of specimens were broken in fabrication with increasing annealing temperatures. This in itself points out the des'-ability of lower stress relief treatments.

Several observations can be made in reviewing this data. The first point is that the tensile strength of the material containing greater than 99% reduction with a 2000°F stress relief is significantly lower than that with 95% reduction, while the other samples in order of decreasing reduction show corresponding decreases in strength, as would be expected. This is attributed to the high annealing temperatures, which on this highly stressed material has resulted in a greater stress relieving effect. Figure 48 shows the effect of annealing temperatures on the tensile properties at the various reductions. The dotted line shows the proposed effect for lower stress relief temperatures on the material containing 99% work. The remaining curves show a direct correlation between reduction and tensile strength. The higher the amount of cold-work, the higher the hardness. However, the more highly strained material recovers at a much lower temperature.

All of the tests regardless of rolling method or stress relief treatment resulted in ductile fracture at 900°F as shown by the elongation values. These values also show that at the lowest stress relief temperature, increasing reductions resulted

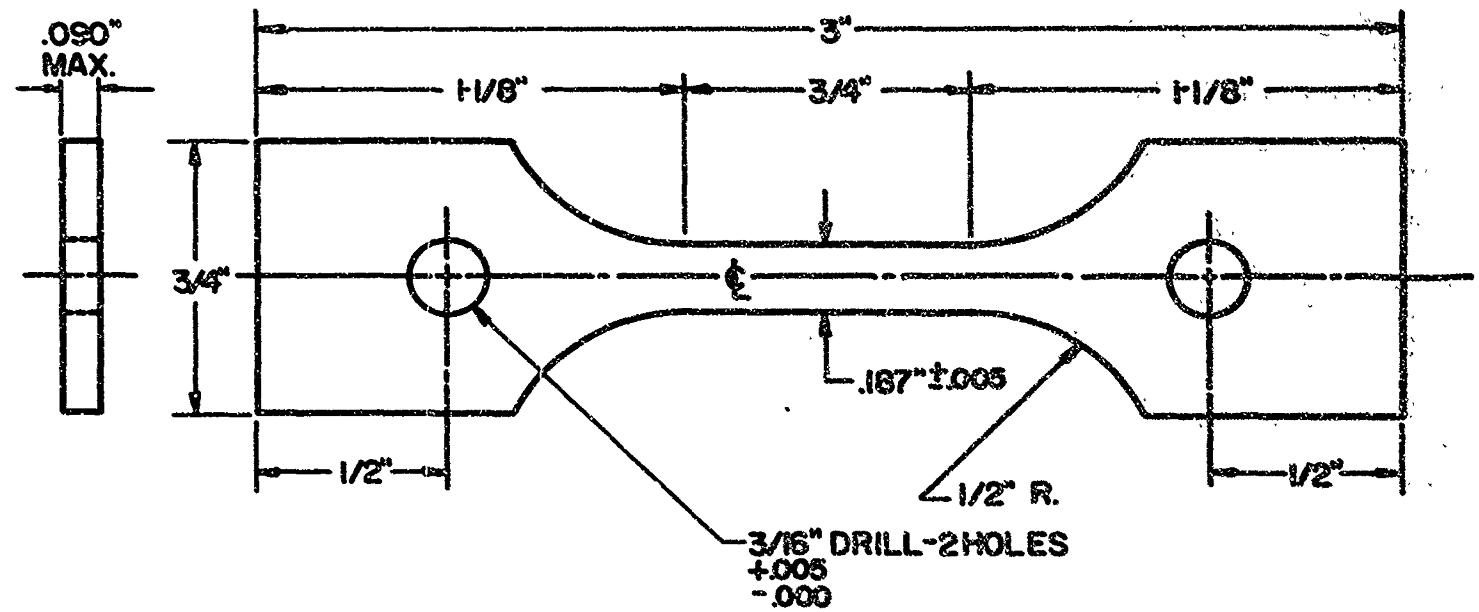


FIGURE 47

L-6S SUBSIZE SHEET TENSILE SPECIMEN

TABLE XXVIII

900°F TENSILE DATA

% Reduction	Identification	Ultimate Tensile Strength 2000	2100	2200	2300	2400	2500	0.2% Yield 2000	2100	2200	2300	2400	2500	% Elongation 2000	2100	2200	2300	2400	2500
99+	A_4A	96.4	90.6	76.1	--	--	--	91.6	88.6	59.3	--	--	--	5.7	6.7	11.7	--	--	--
	B_4A	95.8	91.7	--	--	--	--	90.3	83.5	--	--	--	--	5.5	7.5	--	--	--	--
	C_4A	--	98.7	73.6	--	--	--	--	92.3	53.9	--	--	--	--	7.6	20.1	--	--	--
	D_4A	93.7	--	--	--	--	--	87.9	--	--	--	--	--	3.5	--	--	--	--	--
	E_4A	102.7	--	--	67.3	--	--	100.3	--	--	41.4	--	--	4.9	--	--	26.9	--	--
95	A_1A	108.5	--	68.8	--	--	--	98.6	--	50.5	--	--	--	6.4	--	13.9	--	--	--
	A_1B	113.3	102.8	--	--	--	--	107.5	95.8	--	--	--	--	6.7	8.3	--	--	--	--
	B_1A	105.3	--	--	--	--	--	97.8	--	--	--	--	--	8.0	--	--	--	--	--
	B_1B	104.7	100.5	69.0	57.3	--	--	91.5	90.4	43.3	22.6	--	--	6.3	8.1	26.1	46.7	--	--
	C_1A	--	84.3	87.1	--	--	--	--	82.0	81.5	--	--	--	--	4.3	6.5	--	--	--
	C_1B	108.7	--	--	54.0	--	55.6	93.9	--	--	23.0	--	21.2	7.1	--	--	32.1	--	42.8
92	D_1A	95.9	89.9	75.7	61.3	54.9	50.9	90.9	84.4	68.9	42.9	28.5	21.6	5.5	5.1	8.1	19.6	29.9	44.1
	D_1B	103.6	90.3	72.1	58.5	--	52.4	92.3	85.4	59.7	30.8	--	21.9	5.9	6.8	13.3	28.0	--	42.8
	E_1A	94.7	86.6	82.2	69.8	53.0	48.9	86.8	79.2	55.9	57.0	24.1	18.8	6.3	7.2	19.9	14.0	33.6	41.6
	E_1B	96.3	95.0	67.4	--	--	--	85.7	87.4	48.9	--	--	--	6.3	8.3	22.3	--	--	--
	F_1A	--	91.3	76.0	62.6	--	--	--	84.0	71.2	42.6	--	--	--	6.1	7.3	22.3	--	--
	F_1B	98.0	93.9	81.7	62.7	--	--	92.4	87.8	73.2	39.5	--	--	6.4	7.1	9.1	26.4	--	--
80	B_2A	101.6	--	--	--	--	--	96.1	--	--	--	--	--	6.4	--	--	--	--	--
	B_2B	--	--	--	56.2	--	--	--	--	--	--	--	--	--	--	--	34.4	--	--
	C_2A	104.6	95.0	--	--	--	--	96.2	91.0	--	--	--	--	7.7	7.2	--	--	--	--
	C_2B	96.3	--	69.4	--	--	--	92.0	--	51.7	--	--	--	5.2	--	12.0	--	--	--
	D_2A	83.4	93.4	75.9	64.4	49.0	53.3	81.6	84.3	68.5	42.1	21.8	21.5	4.8	6.3	8.5	19.1	34.0	54.8
	D_2B	--	--	81.8	64.0	55.3	48.2	--	--	70.3	42.3	26.7	23.0	--	--	9.2	24.0	40.5	31.9
	E_2A	96.5	92.3	--	--	--	49.3	91.8	85.3	--	--	--	19.1	5.1	7.5	--	--	--	48.9
	E_2B	99.6	89.5	--	64.6	--	--	94.7	82.3	--	56.6	--	--	7.2	8.9	--	12.0	--	--
	F_2A	95.9	96.8	83.5	--	53.9	52.4	90.4	92.7	73.9	--	22.5	25.0	5.6	6.1	9.3	--	47.2	46.1
	F_2B	98.1	92.4	77.3	--	--	--	94.9	87.4	66.5	--	--	--	5.5	5.2	10.1	--	--	--
60	A_3A	101.7	--	83.8	--	--	--	95.9	--	71.1	--	--	--	5.7	--	10.5	--	--	--
	A_3B	--	91.7	--	--	53.5	--	--	89.4	--	--	41.1	--	--	5.9	--	--	41.2	--
	B_3A	88.5	--	--	--	--	--	81.2	--	--	--	--	--	8.3	--	--	--	--	--
	B_3B	95.4	--	84.4	--	--	--	88.0	--	76.5	--	--	--	7.1	--	10.1	--	--	--
	C_3A	96.5	--	--	--	--	--	92.9	--	--	--	--	--	6.3	--	--	--	--	--
	C_3B	91.7	--	--	--	45.5	--	88.0	--	--	--	19.4	--	6.0	--	--	--	35.5	--
	D_3A	79.2	--	--	68.7	--	--	74.5	--	--	57.3	--	--	6.3	--	--	10.4	--	--
	D_3B	--	84.9	--	69.9	--	--	--	77.5	--	57.6	--	--	--	6.9	--	15.1	--	--
	E_3A	--	83.0	--	--	--	--	--	80.4	--	--	--	--	--	6.9	--	--	--	--
	E_3B	86.4	90.6	81.6	--	--	--	82.3	84.3	77.4	--	--	--	8.5	7.6	10.9	--	--	--
	F_3A	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
	F_3B	86.1	87.8	--	75.4	--	--	81.2	81.5	--	64.5	--	--	7.5	6.8	--	11.9	--	--
40	F_4A	--	--	--	54.8	--	--	--	--	--	--	--	--	--	--	--	27.3	--	--
	A_4B	--	--	--	70.7	--	--	--	--	--	66.0	--	--	--	--	--	9.9	--	--
	B_4B	84.0	--	81.7	73.4	--	--	78.3	--	79.0	69.5	--	--	9.9	--	9.5	9.7	--	--
	C_4B	--	--	--	--	57.4	--	--	--	--	--	43.4	--	--	--	--	--	11.9	--
	D_4B	--	--	68.4	--	--	--	--	--	62.9	--	--	--	--	--	10.7	--	--	--
	E_4B	78.1	71.3	--	65.9	67.1	57.3	73.1	67.2	--	42.8	61.5	51.1	7.9	10.7	--	21.6	8.5	28.7
	F_4B	76.1	78.5	77.1	58.4	--	63.5	71.1	73.2	72.3	29.9	--	54.6	8.5	10.8	7.9	35.6	--	13.4

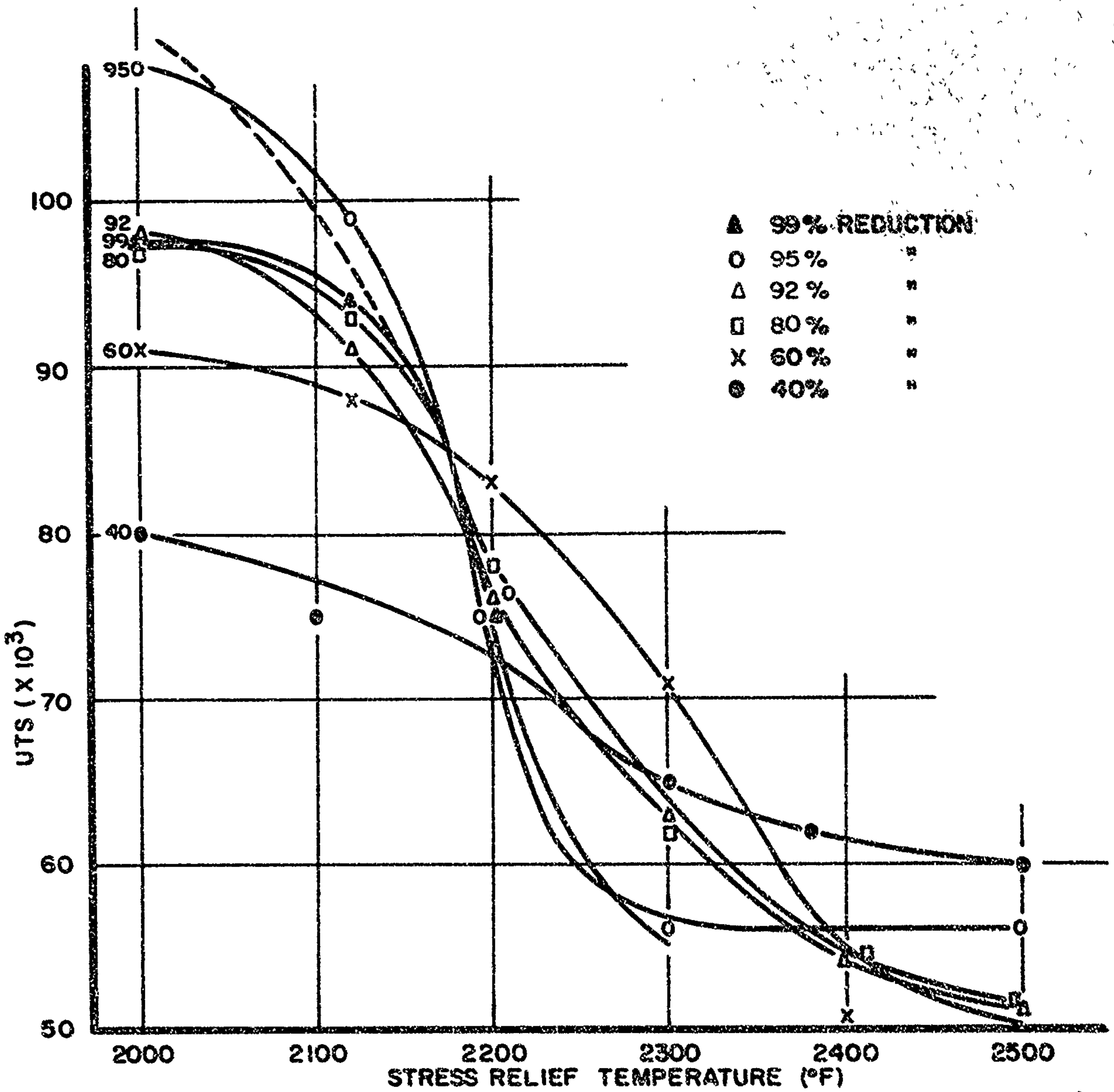


FIGURE 48
EFFECT OF ANNEALING TREATMENTS ON THE ULTIMATE STRENGTH AT VARIOUS REDUCTIONS

in decreasing elongation. As shown in Figure 49, the curves cross each other as the stress relief temperature is increased, resulting in an inverse relationship at the highest stress relief temperature, i.e. elongation increases with increasing reductions.

The tensile properties of the four sheets selected from the bend data are plotted in Figure 50. Note that there is considerable spread in the low temperature annealed strengths. The higher strength of the two "A" sheets is attributed to the lower finish rolling temperature than the "B" sheet from each group.

The elongation values in Figure 51 are also shown to be slightly higher for the lower final rolling temperature.

B. InFab Rolling Studies

1. Sheet Bar Application

From the InFab forging studies discussed under "Ingot Breakdown Evaluation", two forgings were produced from 1-1/2" diameter extruded rounds. The resultant impact forged sheet bar were sectioned into four pieces for InFab rolling evaluation. The rolling schedule for these pieces is given in Figure 52.

2. Rolling Characteristics

a. Initial Rolling

As indicated in the rolling schedule, two pieces were rolled from 1.500" to .250" thick at 3000°F and two pieces at 2400°F. The two pieces rolled from 3000°F resulted in a bond formation between the rolls and the sheet causing a section approximately 2" square to be torn off. This problem was similar to that previously discussed for pack rolling with stainless steel, in that the contact surfaces of the roll and tungsten were clean and incipient melting with subsequent bonding occurred. Other than the bonding problem, the pieces were sound at .250" gauge. The

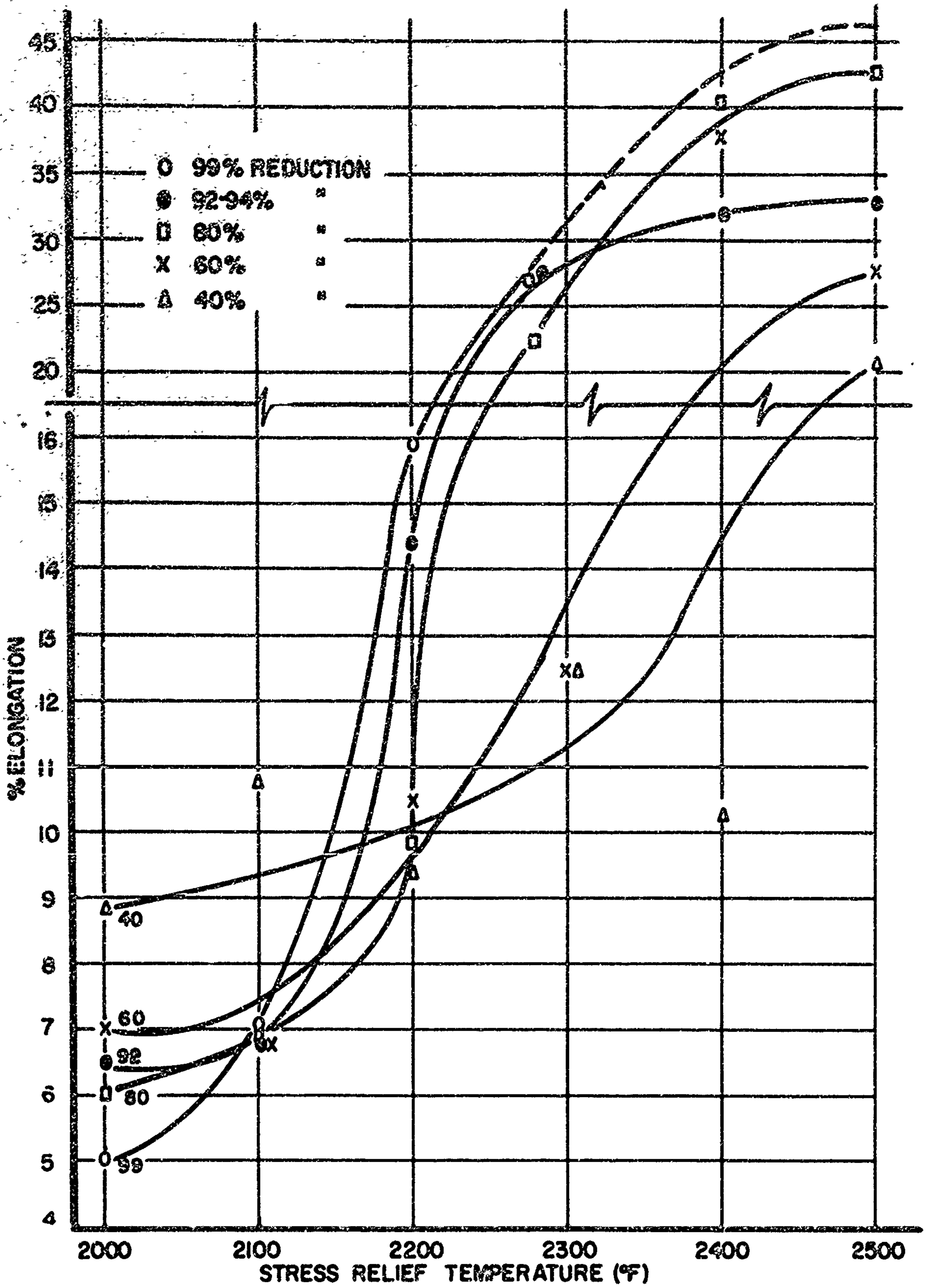


FIGURE 49

EFFECT OF ANNEALING TEMPERATURE ON THE PERCENT ELONGATION AT VARIOUS REDUCTIONS

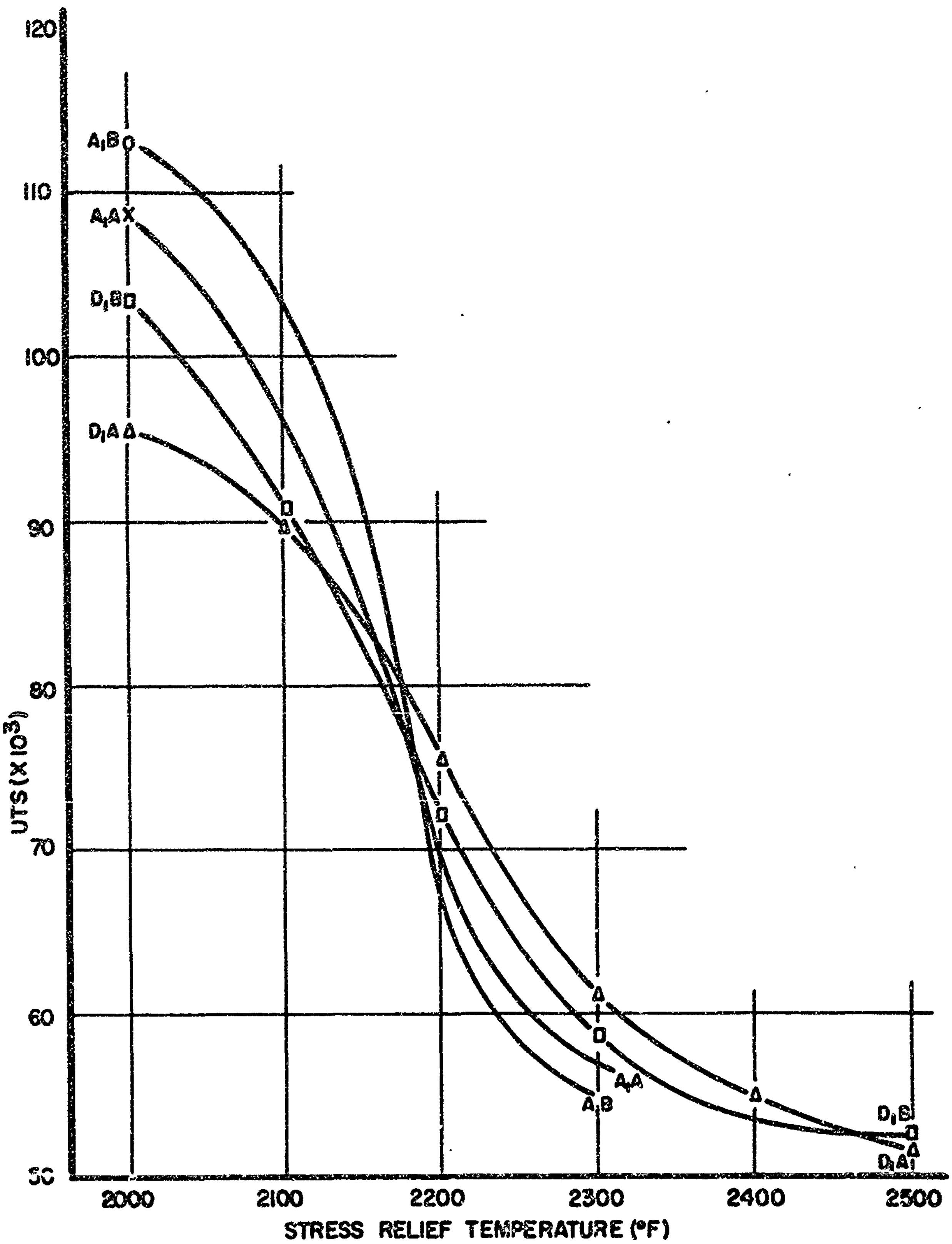


FIGURE 50

EFFECT OF ANNEALING TREATMENTS ON THE TENSILE STRENGTH OF FOUR SELECTED SHEETS

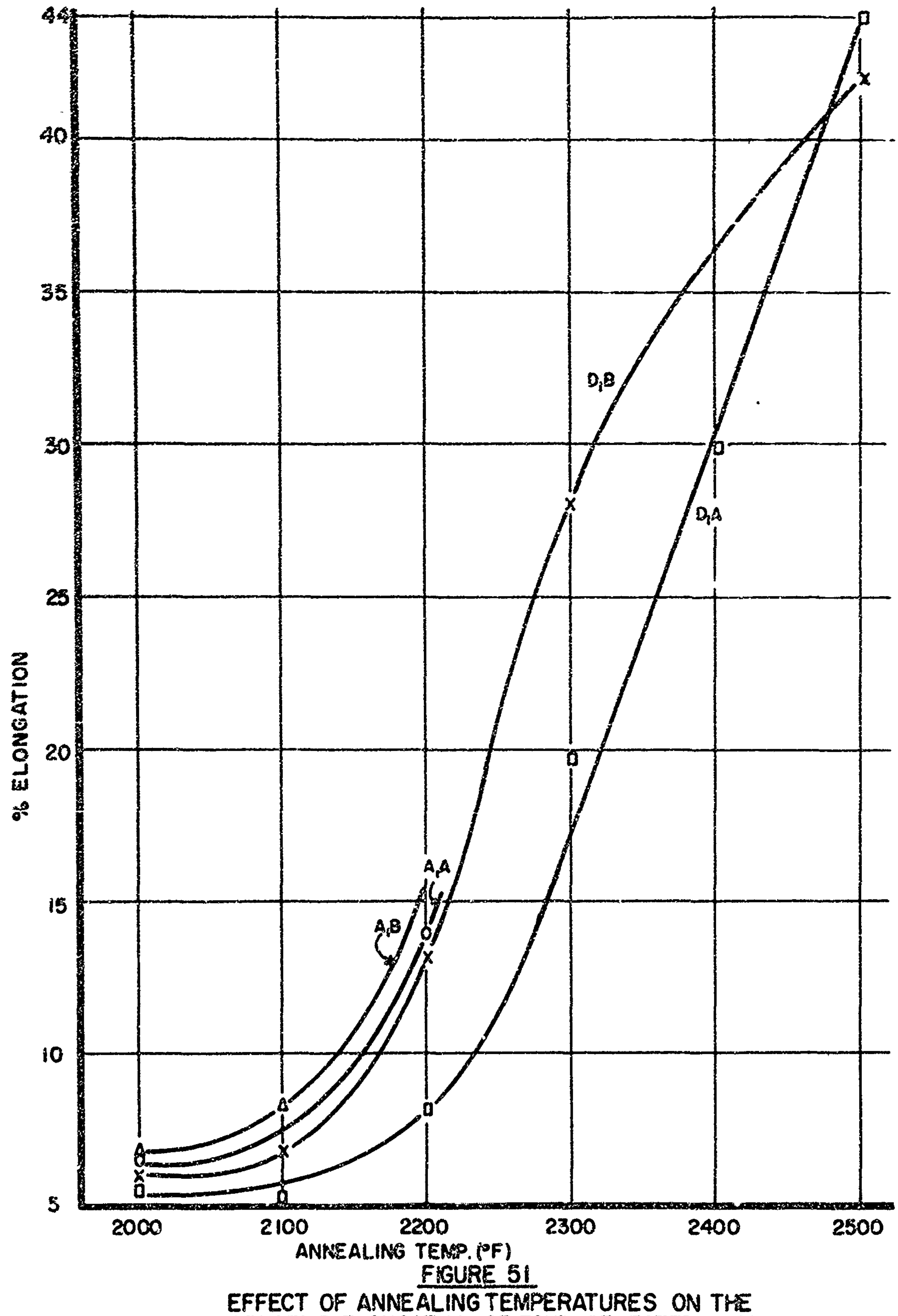


FIGURE 51

EFFECT OF ANNEALING TEMPERATURES ON THE PERCENT ELONGATION OF FOUR SELECTED SHEETS

Figure 52

INFAB ROLLING SCHEDULE

Forged Sheet Bar

(1)	(2)	(3)	(4)
Roll to .250" @ 2400°F	Roll to .250" @ 2400°F	Roll to .250" @ 3000°F	Roll to .250" @ 3000°F
Recrystallize Condition	Recrystallize Condition	Condition	Condition
Roll to .100" @ 2100°F Same Rolling Direction	Roll to .100" @ 2100°F Same Rolling Direction	Roll to .100" @ 2100°F Same Rolling Direction	Roll to .100" @ 2100°F Same Rolling Direction
Condition Roll to .040" @ 1800°F Same Rolling Direction	Condition Cross Roll to .040" @ 1800°F	Condition Roll to .040" @ 1800°F Same Rolling Direction	Condition Cross Roll to .040" @ 1800°F

For Initial Rolling, Reductions Were .075"/Pass - Two Passes/Reheat

For Intermediate Rolling, Reductions Were .050"/Pass - Two Passes/Reheat

For Final Rolling, Reductions Were .025"/Pass - Maximum One/Reheat

remaining two pieces, rolled at 2400°F, appeared to roll satisfactorily; however, post inspection at .250" gauge revealed severe laminations. This problem had not occurred on the conventionally rolled material when rolled in the same temperature range; however, the reduction per pass for the InFab rolling was approximately 30% greater. All pieces, therefore, required conditioning prior to the second rolling operation. Hardness readings on samples of the material rolled at 2400°F indicated 100% recrystallization at 2800°F heat treatment and the two sheets were annealed at this temperature in the InFab furnace.

b. <u>Final Rolling</u>

All of the sheets were rolled from 0.250" to a nominal 0.085" at a constant furnace temperature of 2100°F. At this point, they were conditioned and packed individually between .125" thick stainless steel. Although it was not indicated on the schedule, stainless steel cover plates were used in rolling between 0.085" to 0.040" due to the limiting separating force of the InFab rolling mill. When the first two packs were rolled alternately at 1800°F, severe bonding occurred between the tungsten and the cover plates. In attempting to separate the packs, the tungsten cracked severely, however, sufficient material was available for evaluation.

For the second two packs, the furnace temperature was lowered to 1700°F, however, very slight bonding still occurred. This bonding problem was attributed to the clean surfaces of the tungsten and cover plate material and the contamination-free atmosphere which prevented a protective surface oxide film from forming. The excellent surface condition in areas where bonding did not occur was also attributed to the non-contaminating atmosphere of the InFab enclosure.

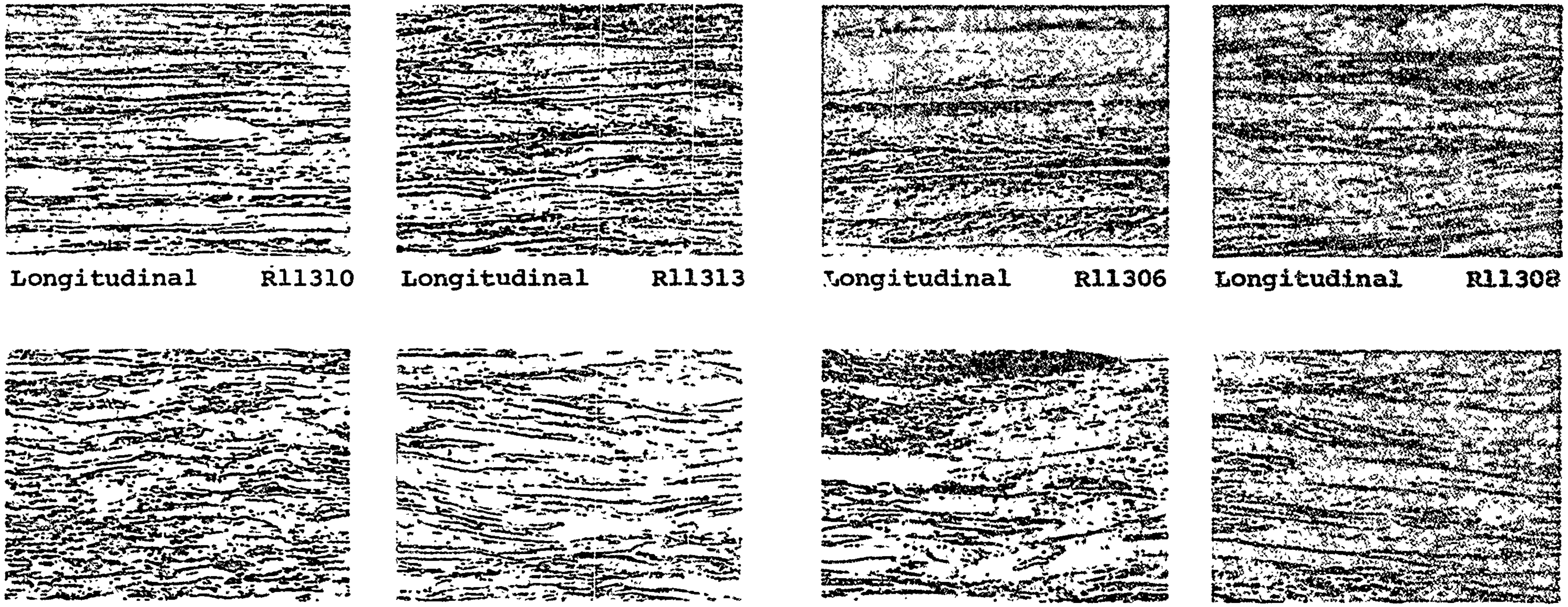
3. Evaluation of Final Rolled Sheet

Evaluation consisted of 1) metallographic observation of the as-rolled structures, 2) determination of the response to heat treatment as determined by metallographic studies, 3) hardness data on the initiation and acceleration of recrystallization, and 4) bend transition determinations.

Hot-cold rolling (2400°F) versus hot rolling (3000°F), and straight versus cross rolling, resulted in significant differences in grain structure as shown in Figure 53. From the material rolled at 2400°F the grain structure of the cross rolled material is wavy and coarser than the straight rolled. Also, the grain boundaries of the longitudinal hot-rolled material are not continuous, which is characteristic of a transverse structure in straight rolled material. The transverse and longitudinal structures of the cross rolled material are consequently shown to be quite similar. The longitudinal hot-cold rolled structure shown is similar to that obtained on conventional rolling utilizing the same general rolling temperatures and reductions.

Figure 53 also shows the as-rolled structures obtained with initial hot-rolling at 3000°F and subsequent hot-cold rolling. It is immediately evident that the hot rolling has resulted in a very coarse grain structure. Except for the coarser structure, the effects of cross rolling are shown to be the same as that previously discussed for hot-cold rolling material.

Samples from all sheets were annealed for one hour at temperatures from 1800° to 2400°F at 100°F increments. The response to these heat treatments as measured by hardness is shown in Figure 54. There is little effect on the hardness drop due to the different rolling techniques. The two hot-cold rolled sheets do show a sudden drop above 2200°F. However, they converge



Longitudinal R11310 | Longitudinal R11313 | Longitudinal R11306 | Longitudinal R11308

Transverse R11311 | Transverse R11312 | Transverse R11307 | Transverse R11309

<u>Straight Rolled</u> — <u>Cross Rolled</u>

Initial Rolling Temperature - 2400°F
Final Rolling Temperature - 1700°F
Constant Gauge - 0.040"
Magnification - 200X

<u>Straight Rolled</u> — <u>Cross Rolled</u>

Initial Rolling Temperature - 3000°F
Final Rolling Temperature - 1700°F
Constant Gauge - 0.040"
Magnification - 200X

Figure 53

As-Rolled InFab Hot-Cold Rolled Microstructures

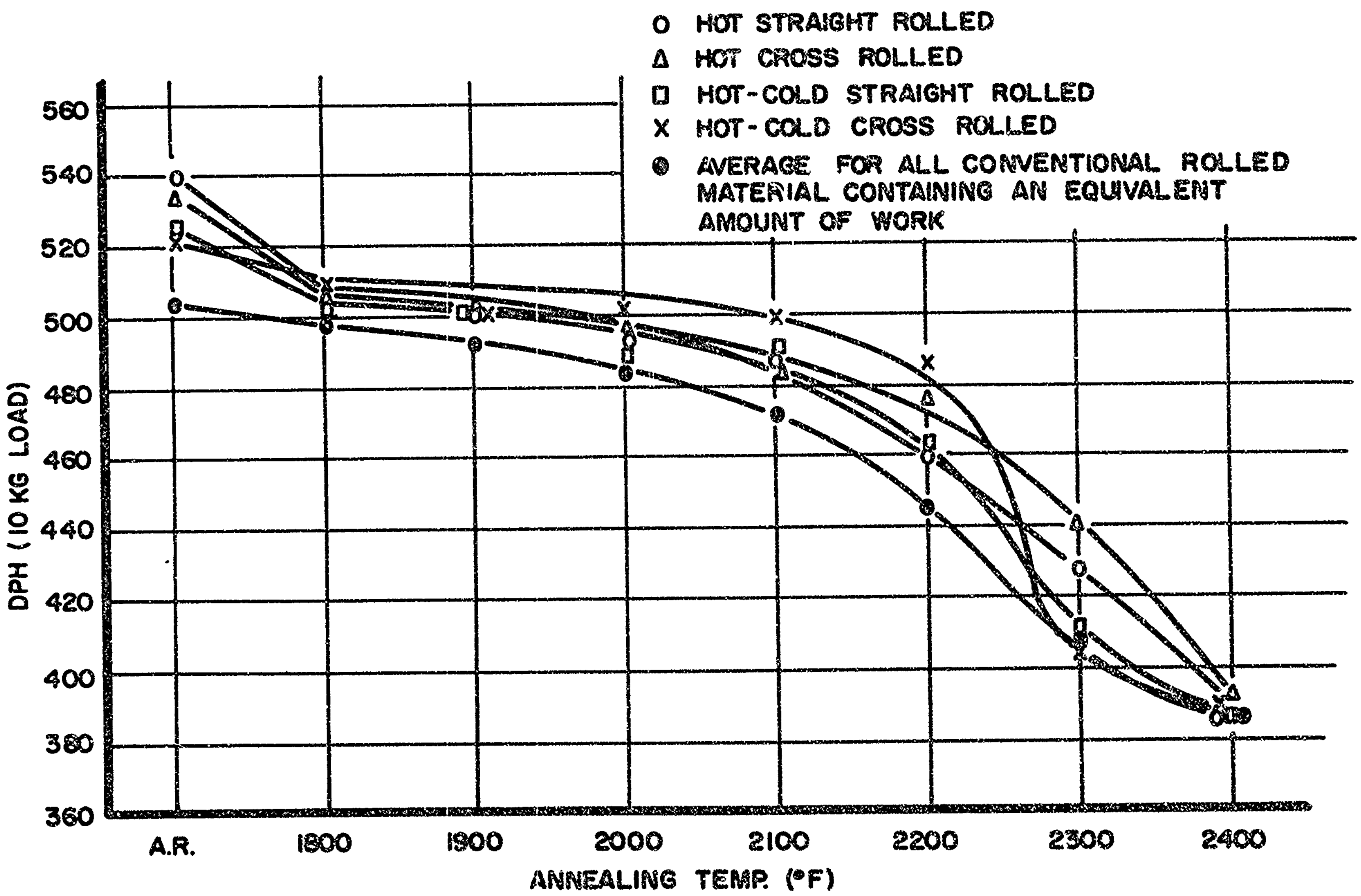


FIGURE 54

EFFECT OF VARIOUS INFAB ROLLING METHODS ON THE RESPONSE TO HEAT TREATMENT

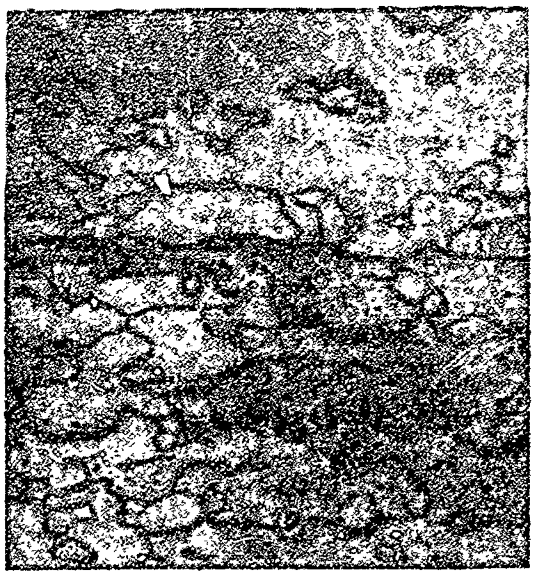
again at 2400°F. This early drop off would indicate a slightly lower recrystallization temperature for the hot-cold rolled material, as would be expected. A plot of conventional rolled material is also shown. No explanation can be given as to why the hardness is lower for this material.

The recrystallization behavior, as determined by microstructural observation, is similar to that previously determined for material conventionally rolled. Figure 55 shows the structure obtained on hot and hot-cold straight rolled material after a one hour 2400°F stress relief as compared to conventionally rolled material after the same heat treatment. The grain size and degree of recrystallization are relatively equivalent for the InFab versus conventional rolling. The grain size of the hot-worked material is slightly larger in both cases although the difference is not as significant as that shown for the as-rolled structures in Figure 53.

Bend transition was determined for all sheets after 1800° and 1900°F stress relief anneals. This data is contained in Table XXIX. The data again points out that true hot rolling is detrimental to bend properties. Cross rolling is shown to have no effect on reducing the transverse bend transition temperature. More extensive rolling and evaluation should disprove this statement based on data obtained on investigations with other materials. The lowest transition temperature compares favorably with the values obtained on conventionally rolled material (approximately 85%). It must be pointed out that this material did not have as much reduction from the last recrystallization anneal as that shown to be optimum (92%) for conventional rolled material. Increased reductions on material rolled in InFab would be expected to show improved bend transition temperatures.

R11315

InFab Hot-Cold Rolled

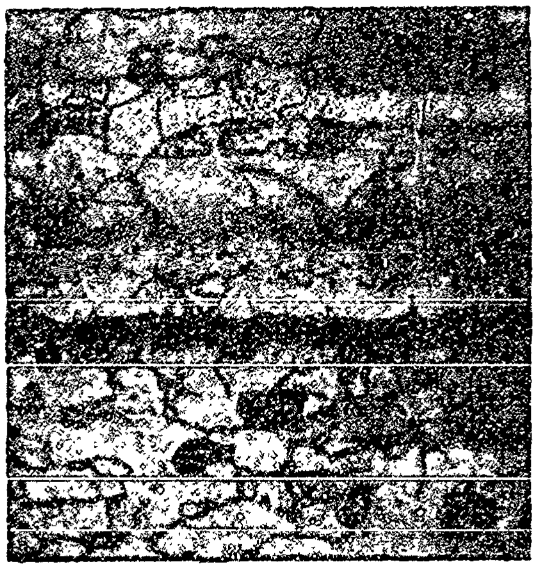


R11314

InFab Hot Rolled

R11031

Conventionally Hot-Cold Rolled



R11033

Conventionally Hot Rolled

<u>InFab Rolling Temperatures</u>

Hot-Cold 2400°-2100°-1800°F
Hot 3000°-2100°-1700°F

<u>Conventional Rolling Temperatures</u>

Hot-Cold 2300°-1900°F
Hot 2700°-1900°F

All Samples Annealed One Hour - 2400°F

Magnification - 200X

Figure 55

Comparison of InFab and
Conventionally Rolled Recrystallized Microstructures

TABLE XXIX

BEND TRANSITION TEMPERATURES - INFAB PROCESSED MATERIAL

Identification	Bend Direction	Rolling Temperature Initial	Intermediate	Final	Bend Transition After Indicated Annealing 1800°F	1900°F
Hot-Cold Rolled	Longitudinal	2400°F	2100°F	1800°F	375°F	400°F
Straight Rolled	Transverse				475°F	500°F
Hot-Cold Rolled	Longitudinal	2400°F	2100°F	1800°F	400°F	400°F
Cross Rolled	Transverse				575°F	500°F
Hot Rolled	Longitudinal	3000°F	2100°F	1700°F	450°F	425°F
Straight Rolled	Transverse				575°F	575°F
Hot Rolled	Longitudinal	3000°F	2100°F	1700°F	450°F	400°F
Cross Rolled	Transverse				600°F	600°F

C. Scale-Up and Refinement of Rolling Practice

1. Investigation of Additional Rolling Variables

a. Sheet Bar Application

As previously discussed, the preliminary rolling investigations did not include several rolling variables which could influence the final properties. The three major variables that had not been investigated were 1) cross rolling, 2) final rolling temperature, and 3) intermediate stress relief annealing. Cross rolling is generally used to promote isotropy within the sheet which is desirable in most applications.

In order to determine the effect of the above variables, the rolling schedule shown in Figure 56 was followed. Sufficient sheet bar was retained to permit rolling of the 24" x 24" sheets necessary for the scale-up required under Phase III of the contract utilizing the optimum schedule determined from the investigations shown in Figure 56.

b. Rolling Characteristics

After initial breakdown of the three sheet bars to .500" thick, samples were heat treated to determine the appropriate stress relief cycle. Figure 57 shows the response to heat treatment as measured by hardness. The three as-rolled pieces were, therefore, annealed for one hour at 2200°F.

These three pieces in addition to the remaining three sheet bars were subsequently all rolled to .110" thick without evidence of cracking. At this point the pieces were segregated for rolling to 0.060" gauge utilizing the various rolling temperatures shown in Figure 56. For all rolling below .110", steel* cover plates

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*AISI-C1095 used for 1250° and 1550°F rolling temperature
AISI-301 stainless used for 1850°F rolling temperature

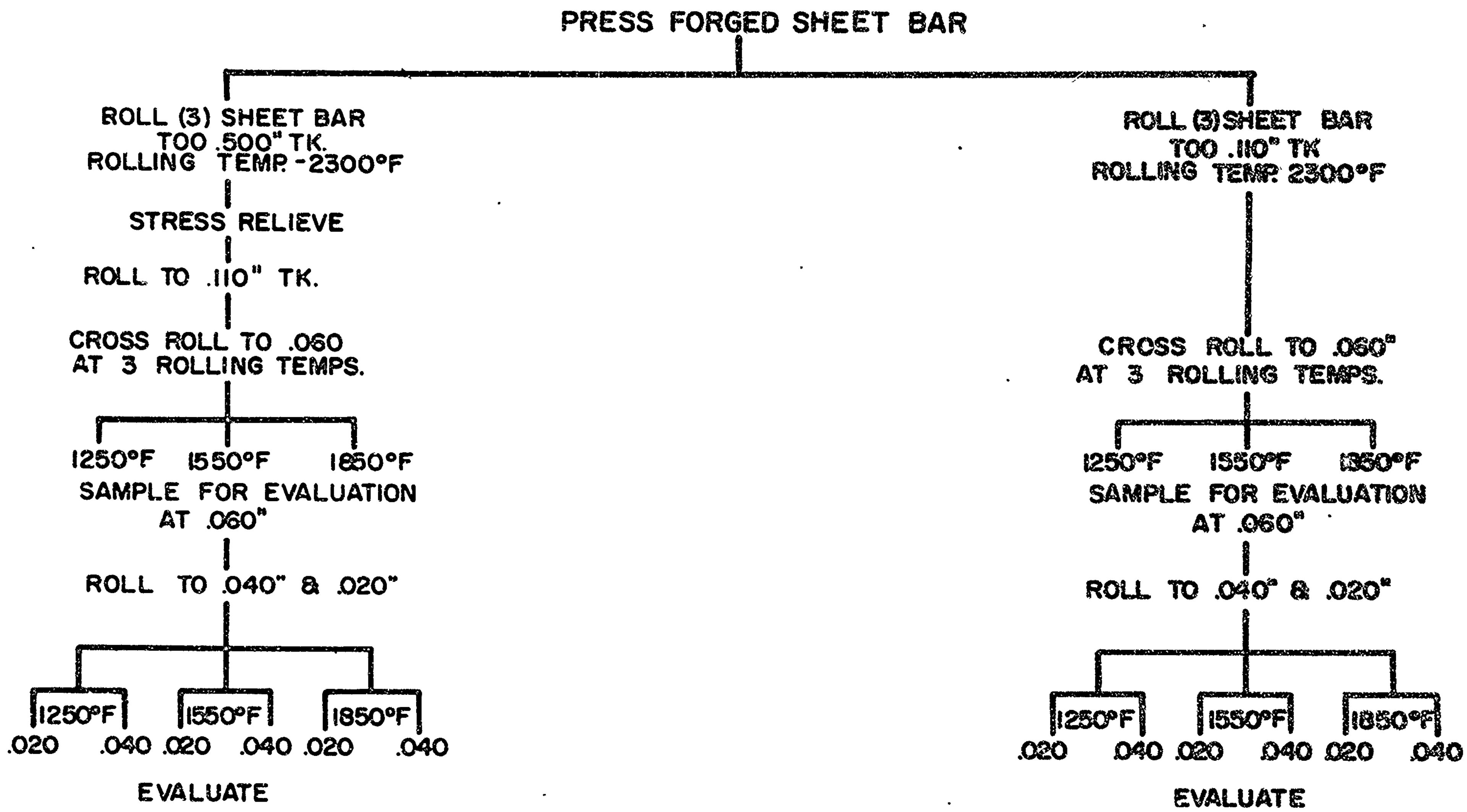


FIGURE 56

SECOND INVESTIGATIVE ROLLING SCHEDULE

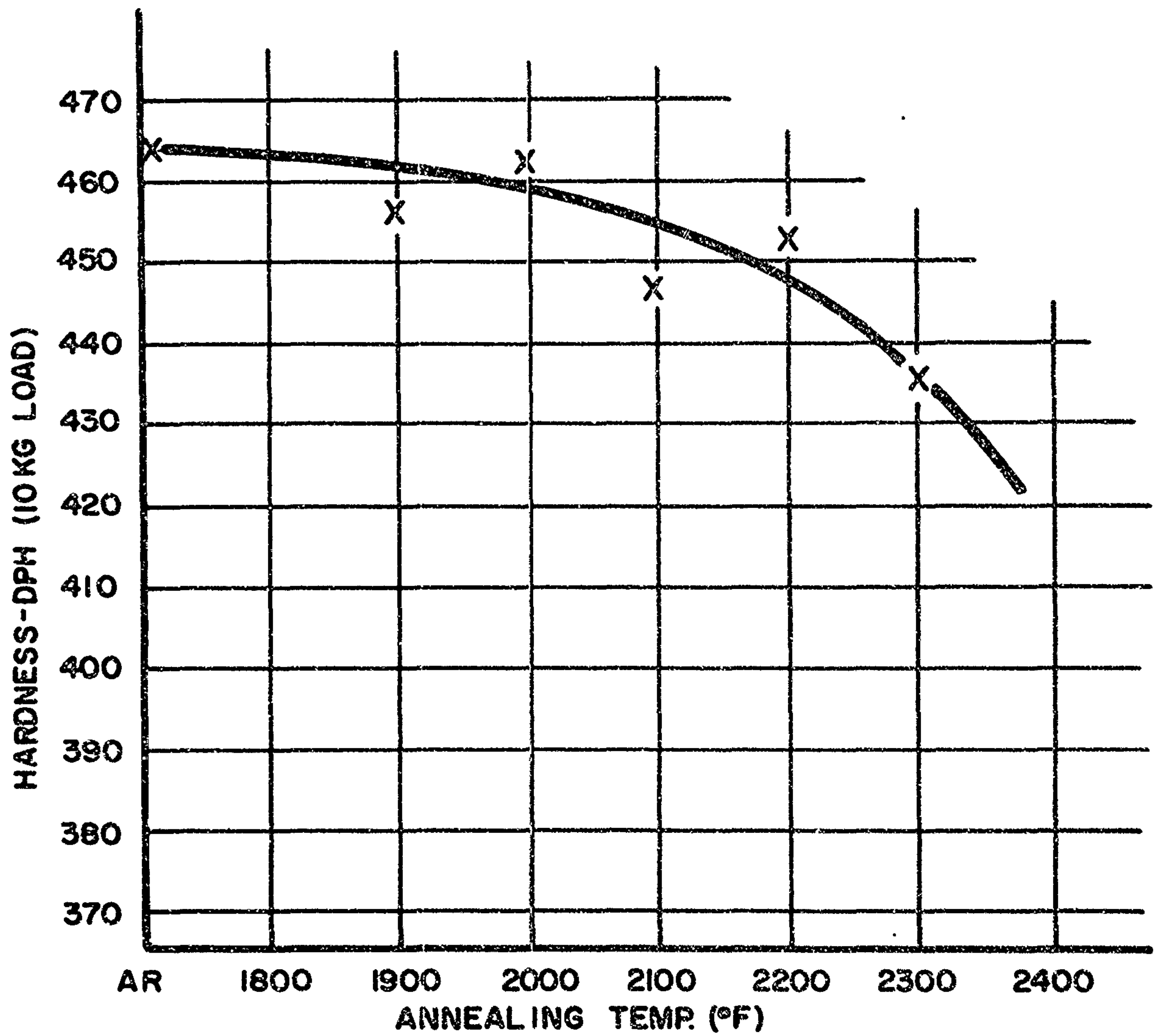


FIGURE 57

HARDNESS RESPONSE TO HEAT TREATMENT
ON .500" MOLDOUT — 1 HOUR CYCLE

were used to permit reduction to the desired gauge. The cover plates also helped to maintain heat within the tungsten. One problem associated with the cover plates is edge cracking which occurs when the edge of the tungsten slips out of the cover plates during rolling. The resulting non-uniform reduction causes edge tears. Figure 58 representing typical .040" gauge material, shows no edge cracking.

c. Evaluation of Final Rolled Sheet

(1) Flattening and Descaling

Following the final rolling operations, all sheets were roller leveled after preheating to 1400°F. All pieces were black (below 1100°F) upon exiting from the leveler. The .020" gauge material cooled below red heat (1100°F) before entering the leveler. The results, after flattening, showed that the maximum deviation from flatness as measured by MAB recommendations (Report 176-M) was 3%. The maximum permissible variation under this specification is 4%.

All sheets were descaled in molten caustic and acid baths to remove the oxide prior to cutting test specimens. Test specimens were cut from each sheet according to the plan shown in Figure 59.

(2) Reduction and Annealing Treatments

The starting sheet bars were the same gauge regardless of final gauge produced. The in-process stress relief anneals were also accomplished at a constant gauge regardless of final sheet gauge. By using this technique, various reductions existed in the final sheet in relation to the annealing temperatures and thus a correlation of properties was expected. The reductions encountered in each gauge sheet are as follows:

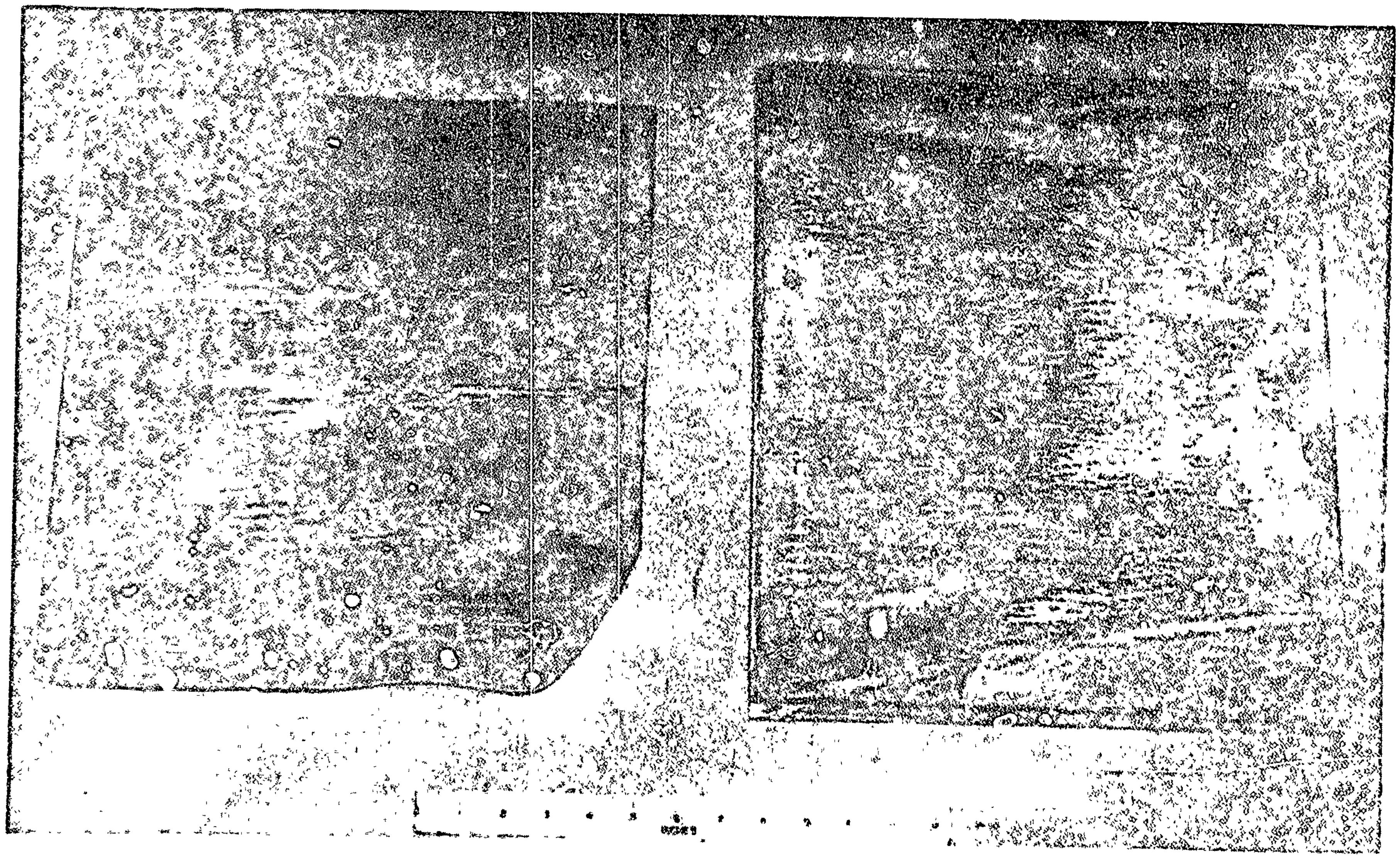


Figure 58

As-Rolled .040" Sheet
Rolling Temperature 1550°F

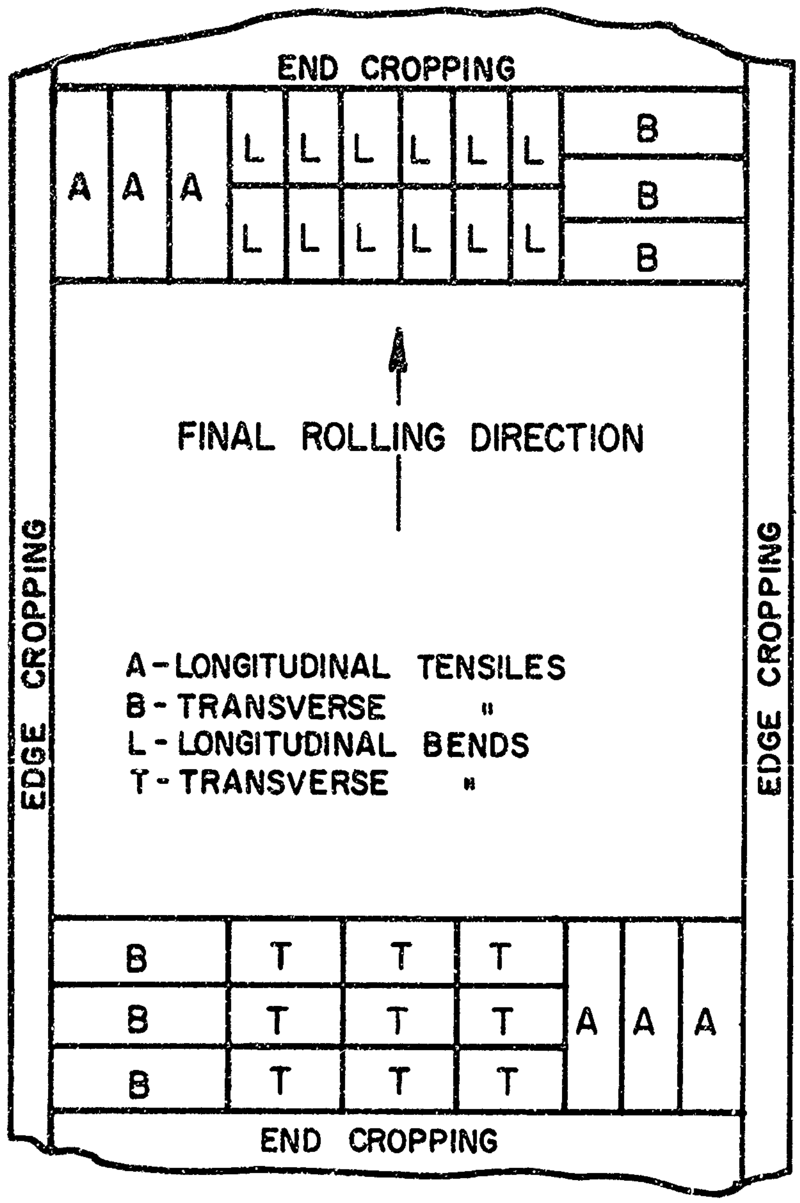


FIGURE 59

TEST SI 'EN CUTTING PLAN

.060" Sheet

95% - Total reduction from sheet bar

88% - Reduction from intermediate stress relief

.040" Sheet

96% - Total reduction from sheet bar

92% - Reduction from intermediate stress relief

.020" Sheet

98% - Total reduction from sheet bar

96% - Reduction from intermediate stress relief

(3) Cross Rolling Ratio

Cross rolling was initiated at .110" regardless of the final gauge produced. This resulted in increasing degrees of cross rolling as the gauge was decreased. The determining formula and the actual ratios for the three final gauges are as follows:

$$\text{Cross Rolling Ratio} = \frac{\text{\% Reduction Before Crossing}}{\text{\% Reduction After Crossing}}$$

.060" Sheet

Cross Rolling Ratio - 1.95/1 Actual or 2/1 Nominal

.040" Sheet

Cross Rolling Ratio - 1.45/1 Actual or 1.5/1 Nominal

.020" Sheet

Cross Rolling Ratio - 1.09/1 Actual or 1/1 Nominal

(4) Response to Heat Treatment

Eighteen sheets were final rolled to accommodate the rolling schedule given in Figure 56. Samples from each of the eighteen sheets were subjected to one hour heat treat-

ments over the range of 1600° to 2400°F. Figures 60, 61, and 62 show the hardness response to these anneals. The intermediate stress relief and final rolling temperatures had very little effect on the response to heat treatment. The curves in Figure 60 for the .060" material are the only ones in which even a slight effect due to rolling temperatures can be observed. This figure indicates that a rapid drop in hardness initiates earlier in the material with the lowest rolling temperature (note hardness at 2100°F); the remaining curves are in direct correlation with their respective rolling temperatures. Also, at the highest temperature checked (2400°F), the relationship still exists; that is, the lowest rolling temperatures result in a lower complete recrystallization temperature and correspondingly the highest rolling temperature has the least degree of recrystallization at the same annealing temperature. The remaining curves in Figures 61 and 62 are so close that no definite trends can be established. In comparing all three curves, it is shown that the initiation of recrystallization or rapid hardness drop starts at 2100°F for 0.060", 2000°F for .040", and 1950°F for .020" gauge. This was to be expected based on increasing total reduction as the gauge decreased.

A secondary investigation was initiated to determine 1) the hardness response to heat treatments for various annealing times and 2) the effect of time and temperature on the recrystallized grain size. Annealing treatments were run over the range of 1800° to 2700°F at 100°F increments. The times at temperature were 5, 10, 20, 30, and 60 minutes. Figure 63 shows the hardness response plotting constant time versus temperature. In the range of 2200°F, the hardness is starting to decrease rapidly and a direct correlation between annealing time and hardness exists. Approaching complete recrystallization, the hardness values become more erratic; however, at 2500°F the correlation still exists. As

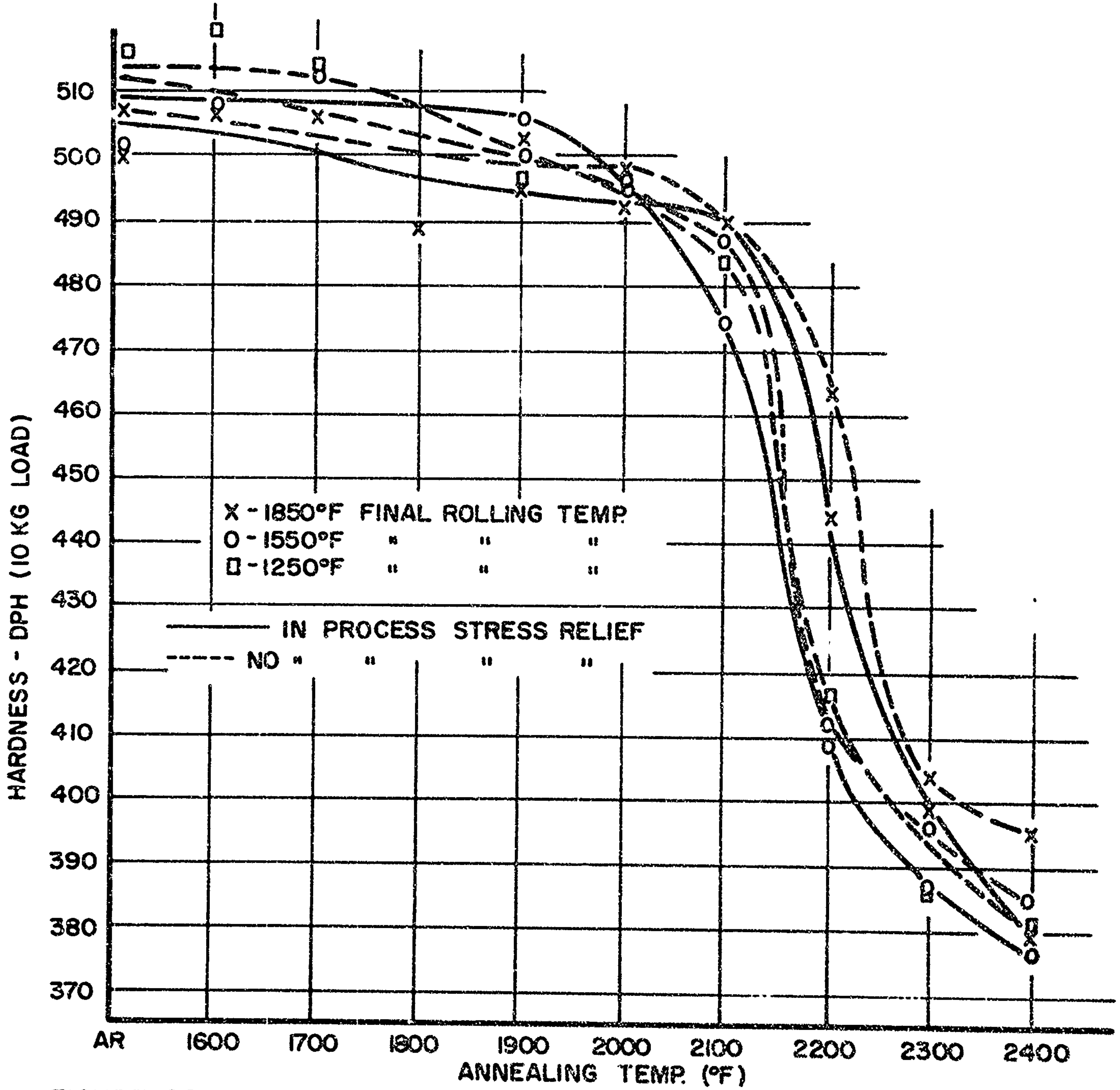


FIGURE 60— HARDNESS RESPONSE TO HEAT TREATMENT OF .060" SHEET

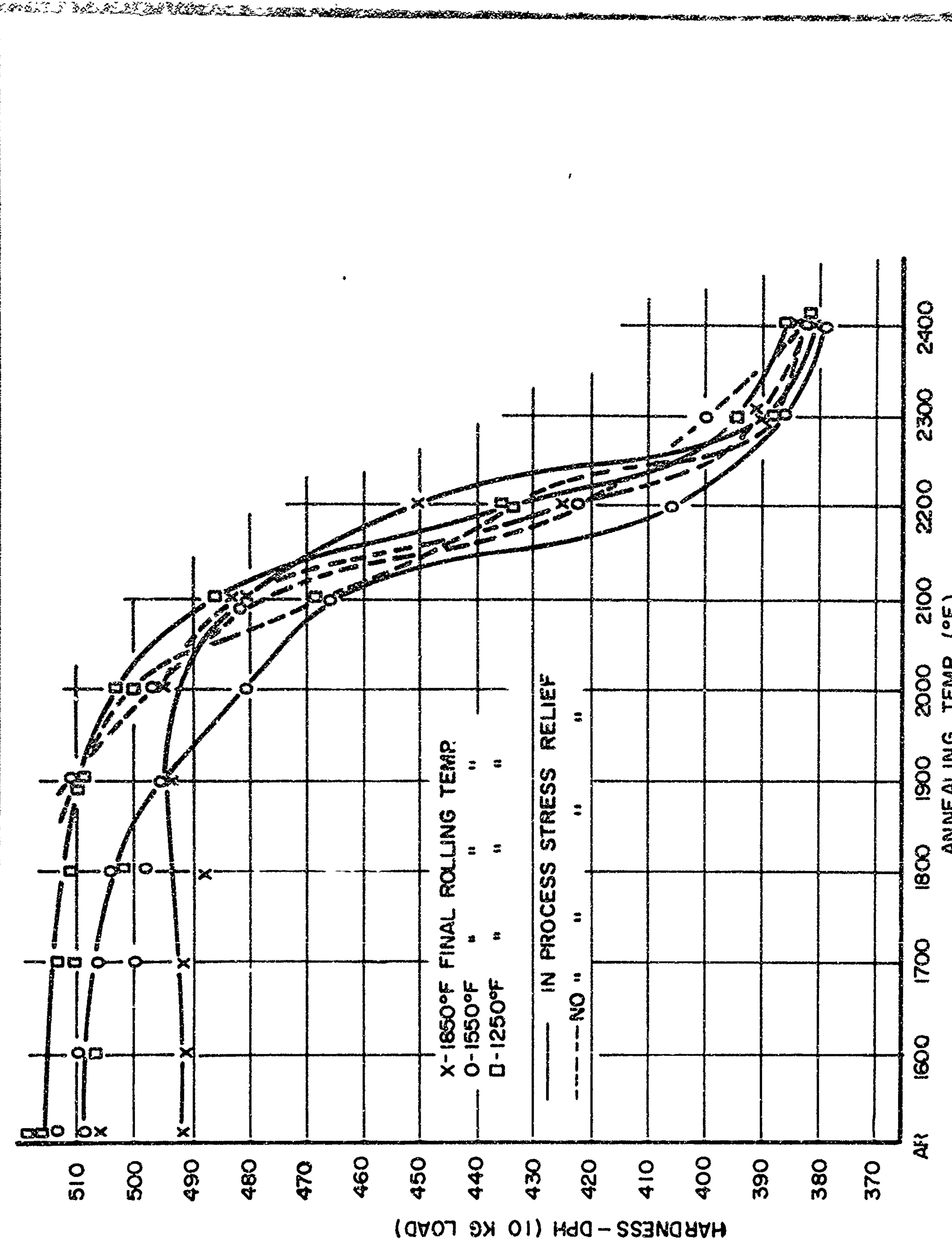


FIGURE 61--HARDNESS RESPONSE TO HEAT TREATMENT OF .040" SHEET

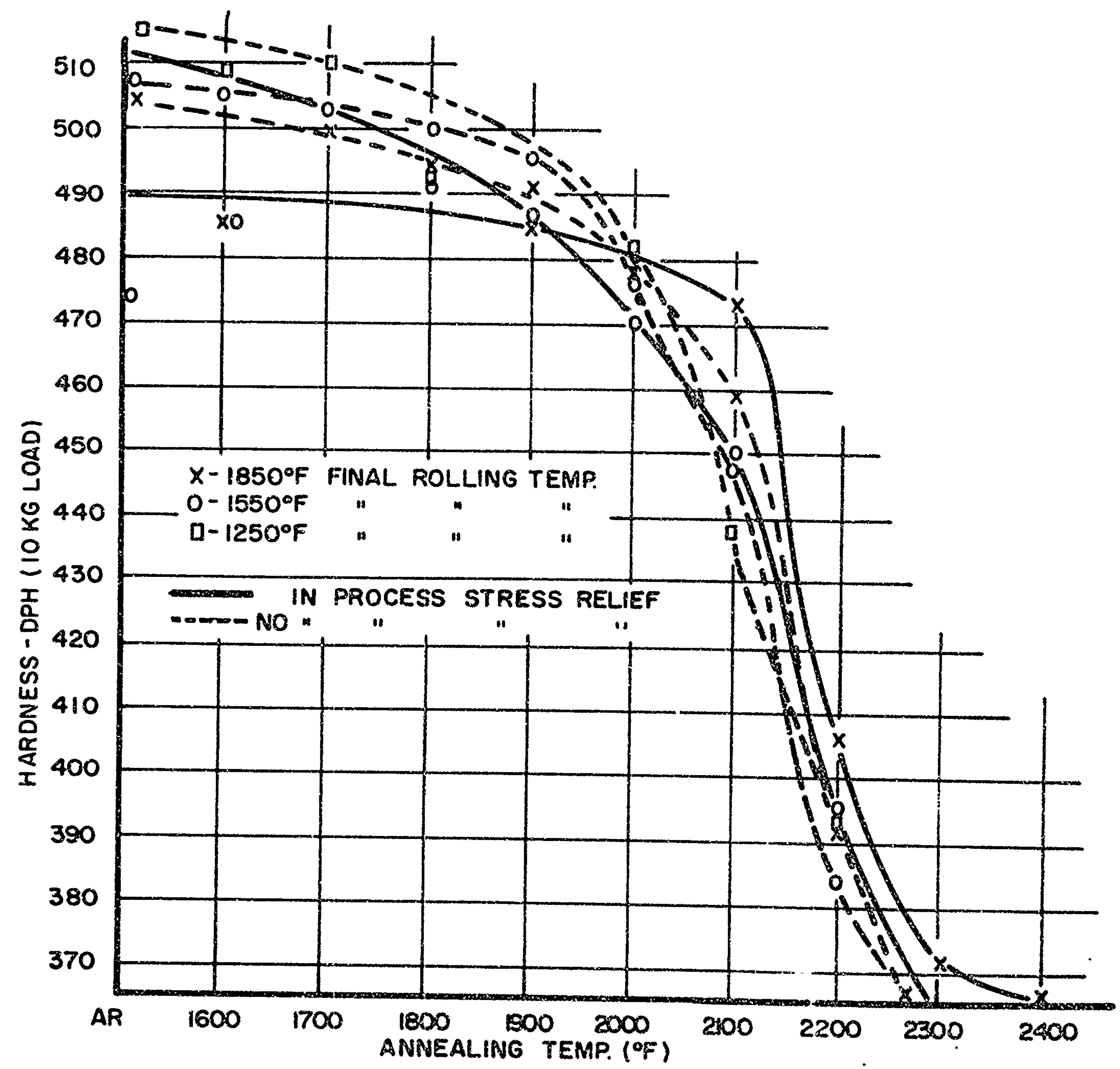


FIGURE 62— HARDNESS RESPONSE TO HEAT TREATMENT OF .020" SHEET

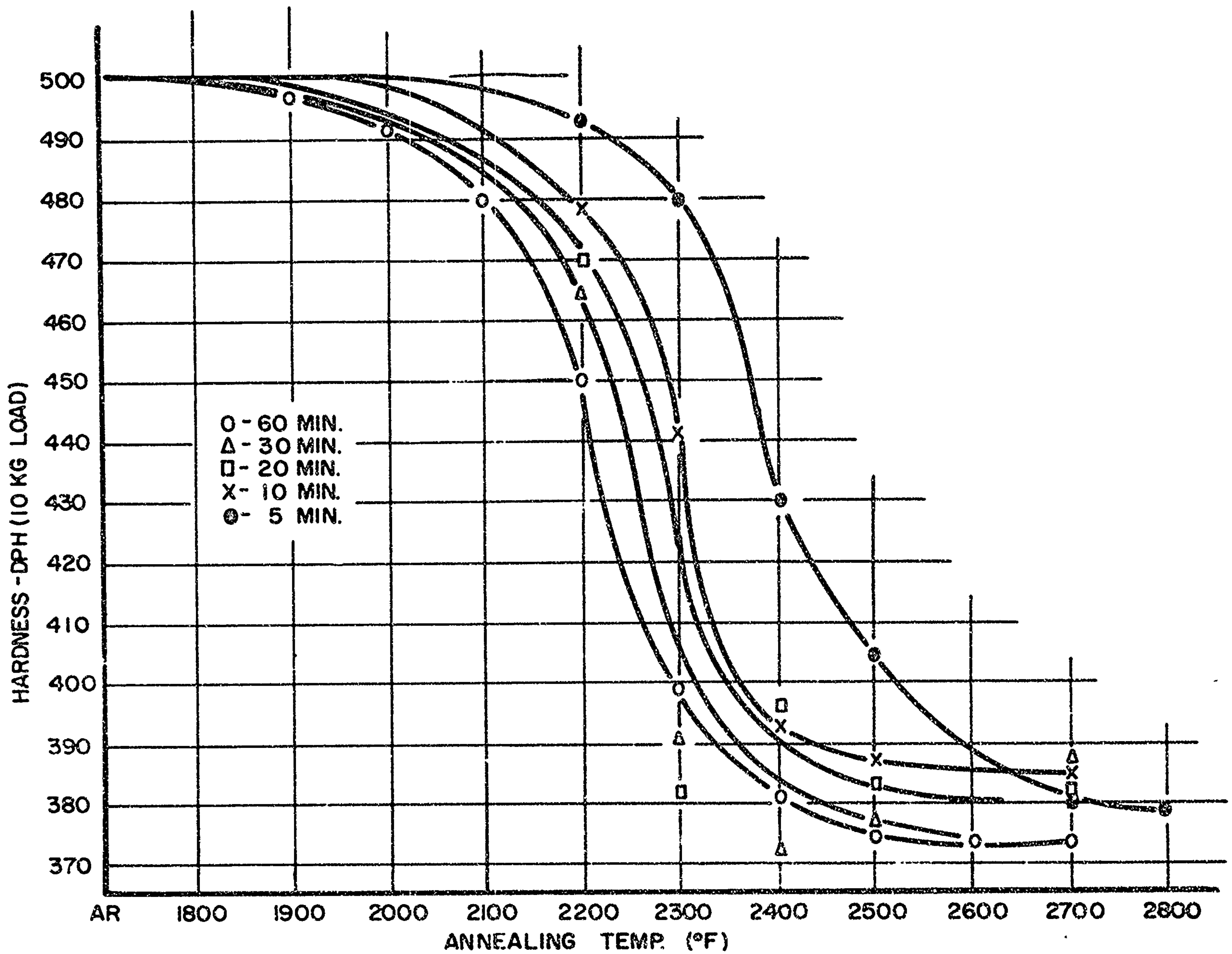


FIGURE 63

HARDNESS RESPONSE TO VARIOUS TIME—TEMP. HEAT TREATMENTS

shown in Figure 64, the difference between the estimated 100% recrystallization temperature for five and sixty minutes is 200°F. This figure also shows that there is little or no difference in the resulting recrystallized grain size due to the various time-temperature heat treatments.

(5) Tensile Properties

Transverse tensile tests were conducted on each sheet. Based on previous work, the stress relief temperatures of these specimens were limited to 1600°, 1700°, and 1800°F. Tensile test procedures, in accordance with MAB 176-M, were as follows:

Test Temperature - 900°F

Strain Rate - .005"/in/min to 0.6% yield
.05"/in/min to fracture

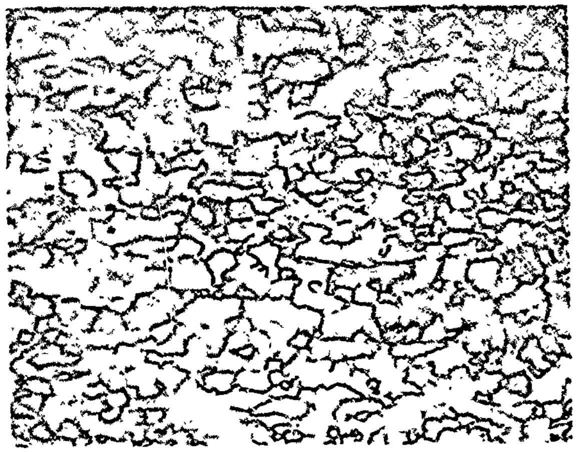
Exception was taken to the specimen size with the following used:

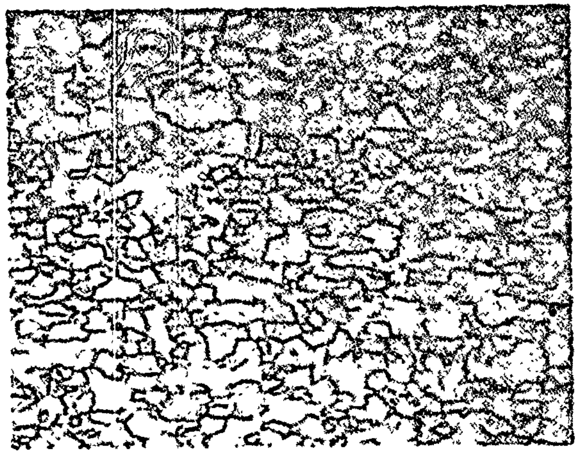
Gauge Length - 3/4"

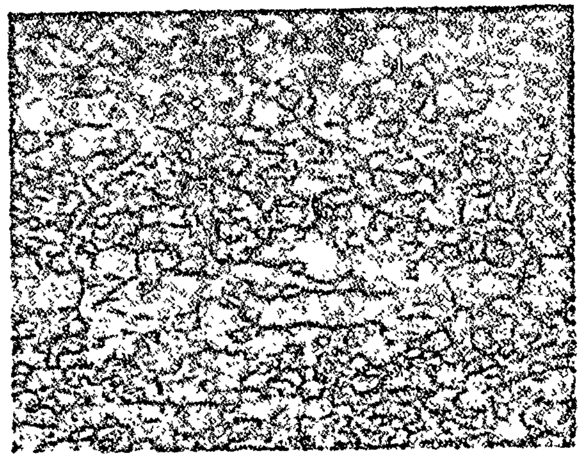
Gauge Width - .187"

Figures 65, 66, and 67 are plots of ultimate strength and elongation in relation to the final stress relief temperature. It is shown that a significant effect on the tensile properties results from these relatively low stress relief temperatures. In reviewing only the ultimate strength curves in all three figures, it is shown that a distinct correlation between final rolling temperature and strength exists. In every case, the strength is shown to be increased sharply with decreasing rolling temperatures.

Trends based on elongation are more indiscrete. In Figure 65, it is shown, with one exception, that the elongation increases with increasing stress relief temperature.


5 Minutes at 2800°F R11699


10 Minutes at 2700°F R11698


20 Minutes at 2700°F R11697


30 Minutes at 2600°F R11696


60 Minutes at 2600°F R11695

Figure 64

Effect of Annealing Time on the Recrystallized Grain Size
0.040" Thick Vacuum Heat Treatment
Temperature Shown is Minimum for Estimated 100% Recrystallization
Magnification 100X

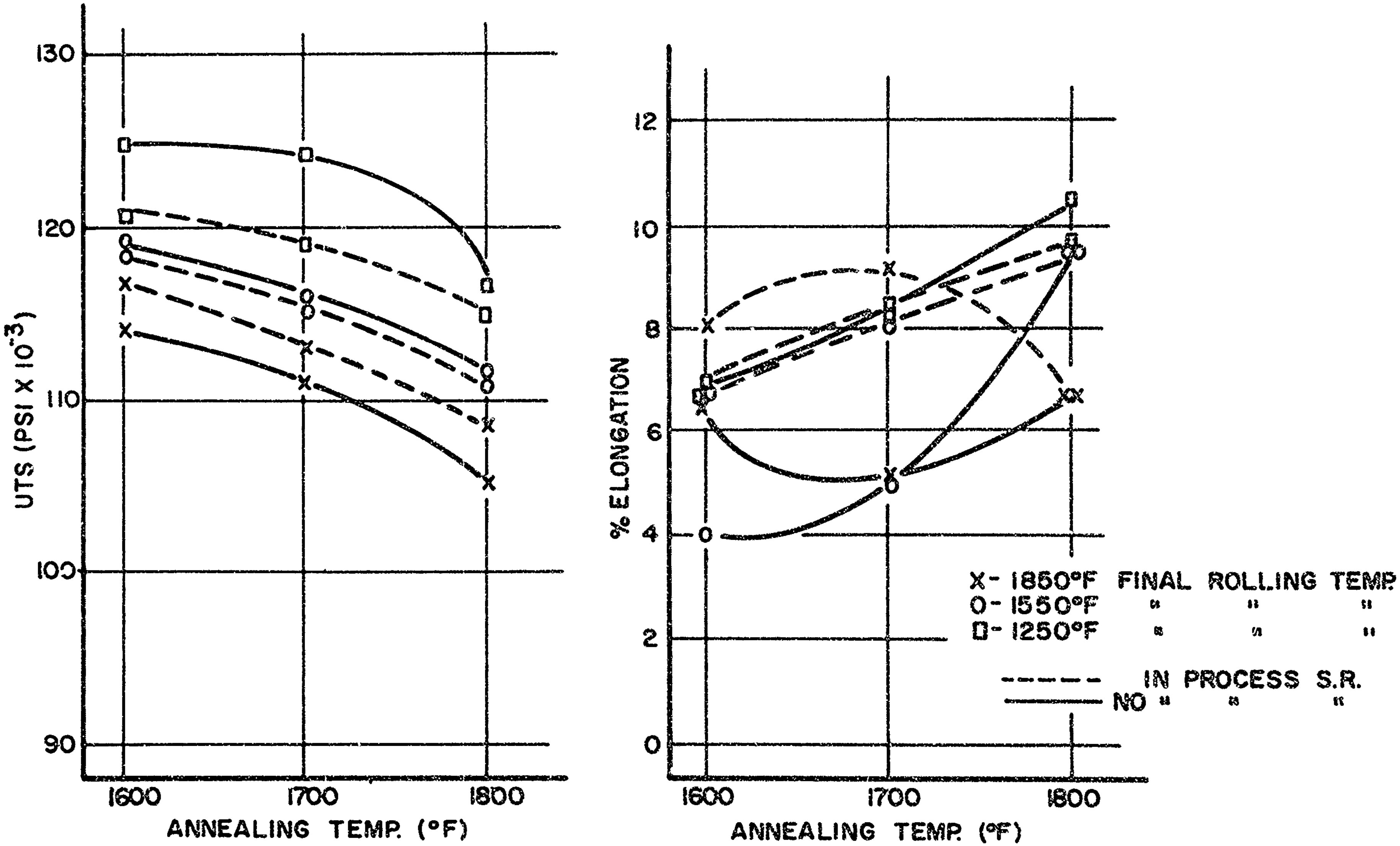


FIGURE 65

TRANSVERSE TENSILE PROPERTIES OF .060" SHEET
TEST TEMP. — 900°F

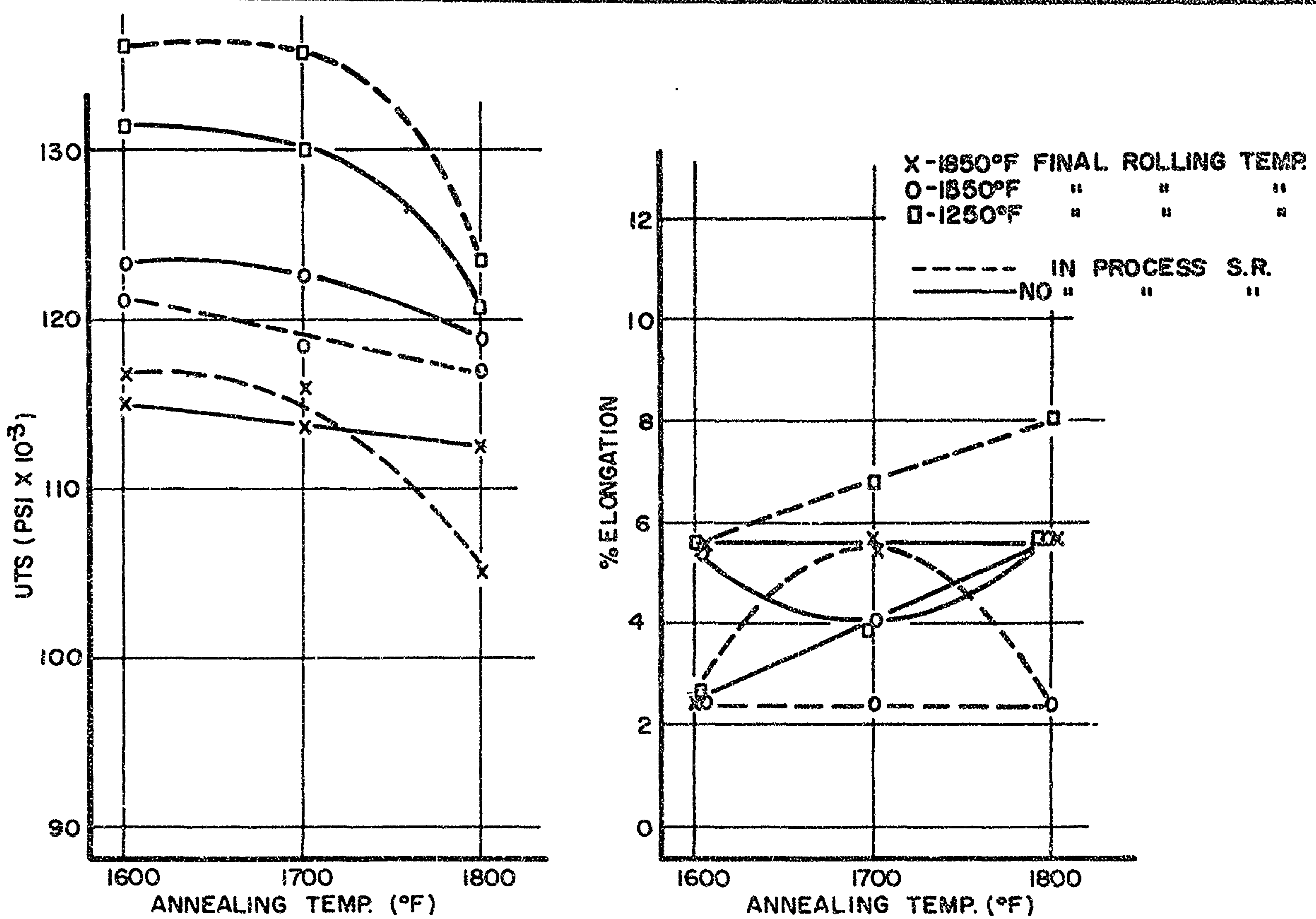


FIGURE 66

TRANSVERSE TENSILE PROPERTIES OF .040" SHEET
TEST TEMP.—900°F

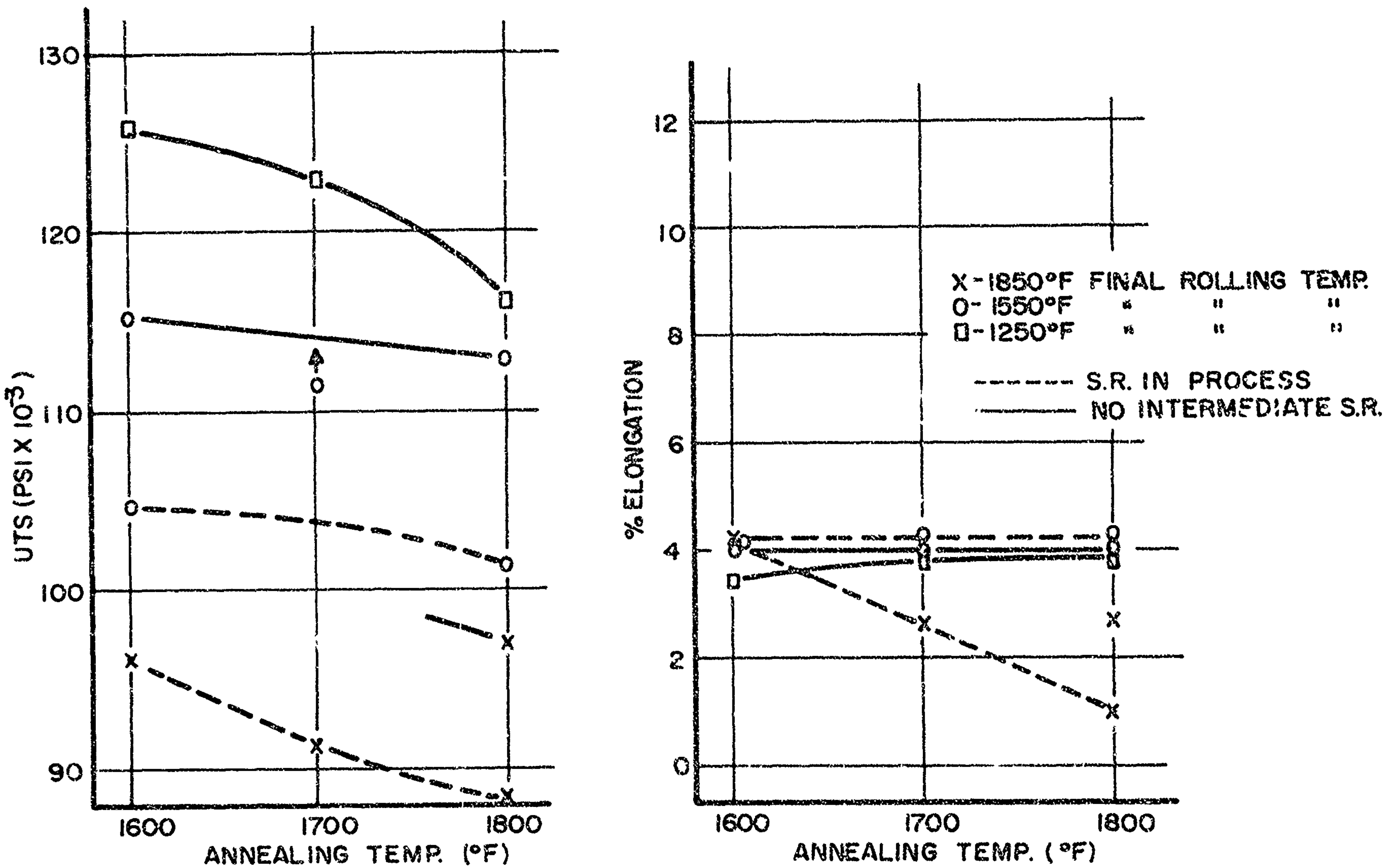


FIGURE 67

TRANSVERSE TENSILE PROPERTIES OF .020" SHEET
TEST TEMP.—900°F

From this figure, it could also be said that the in-process stress relief improves the final elongation values. In reviewing all three figures, it is shown that as the gauge decreased, the average elongation values are typically decreasing. It is suggested that the increasing degree of cold rolling in the light gauges also contributed to the lower elongation.

The lower relative strength values for the .020" material in Figure 67 are indicative of lamination and machining problems in specimen preparation. This may also have contributed to the low elongations on this material.

(6) <u>Bend Transition Properties</u>

Figures 68, 69, and 70 are plots of the transverse bend transition temperature versus the three stress relief temperatures investigated. In Figure 68, representing .060" material, two sheets show significantly better results than the remaining four. Both of these had an in-process anneal and the final rolling temperatures were 1250° and 1550°F, respectively. The same process, except for a final rolling temperature of 1850°F, is shown to have a much higher transition temperature. The lowest transition temperature for .060" material was 225°F.

In Figure 69, representing the .0 0" material, three sheets show equally low transition temperatures at one or more of the final stress relief temperatures. Two of these three received an in-process anneal and, as in the .060" material, the final rolling temperatures of 1250° and 1550°F were better than 1850°F. The lowest transition temperature for .040" material was 200°F.

Transition temperatures for the .020" material are shown in Figure 70. One sheet was so susceptible to

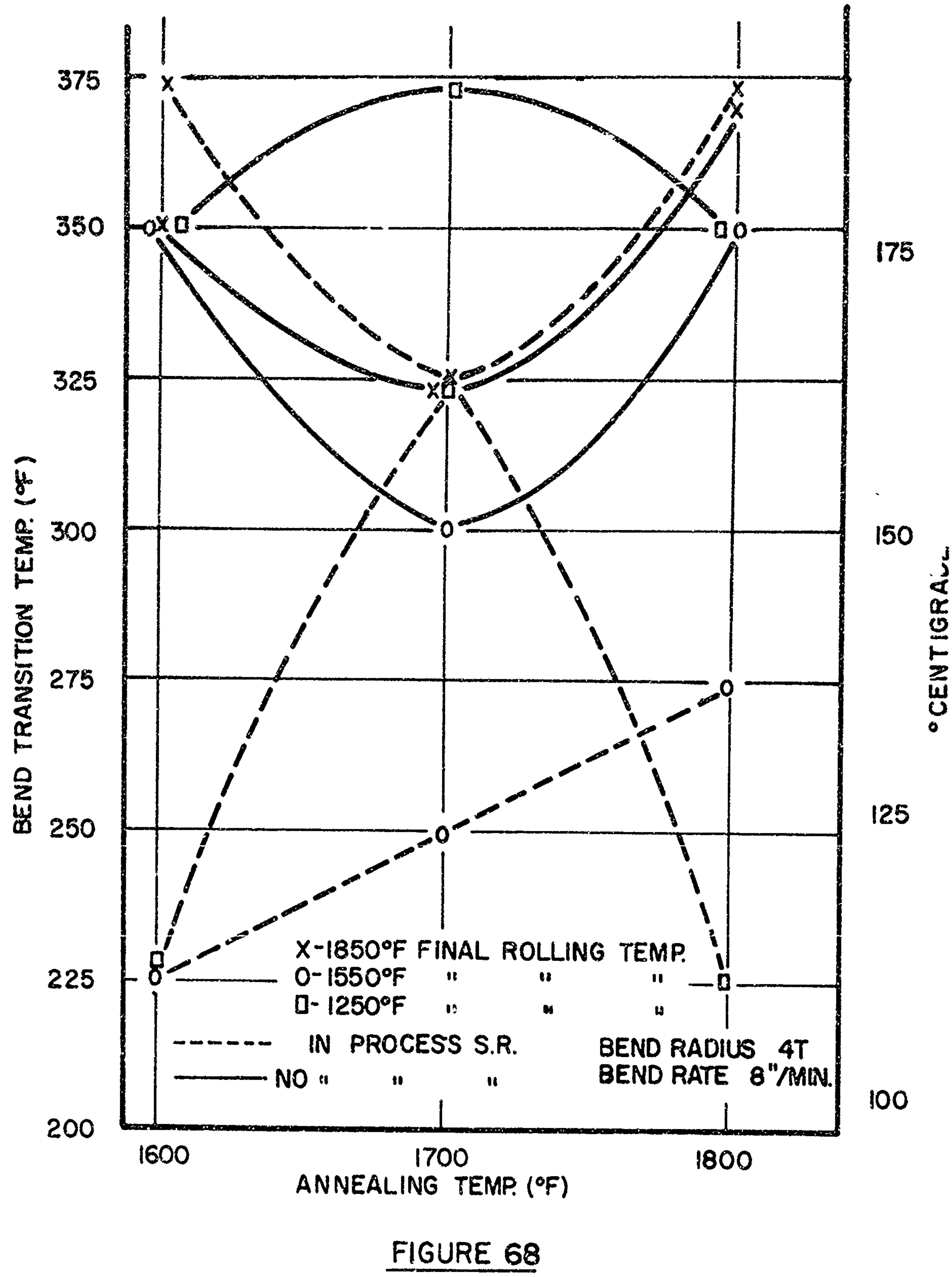


FIGURE 68

TRANSVERSE DUCTILE BRITTLE BEND TRANSITION OF .060" SHEET

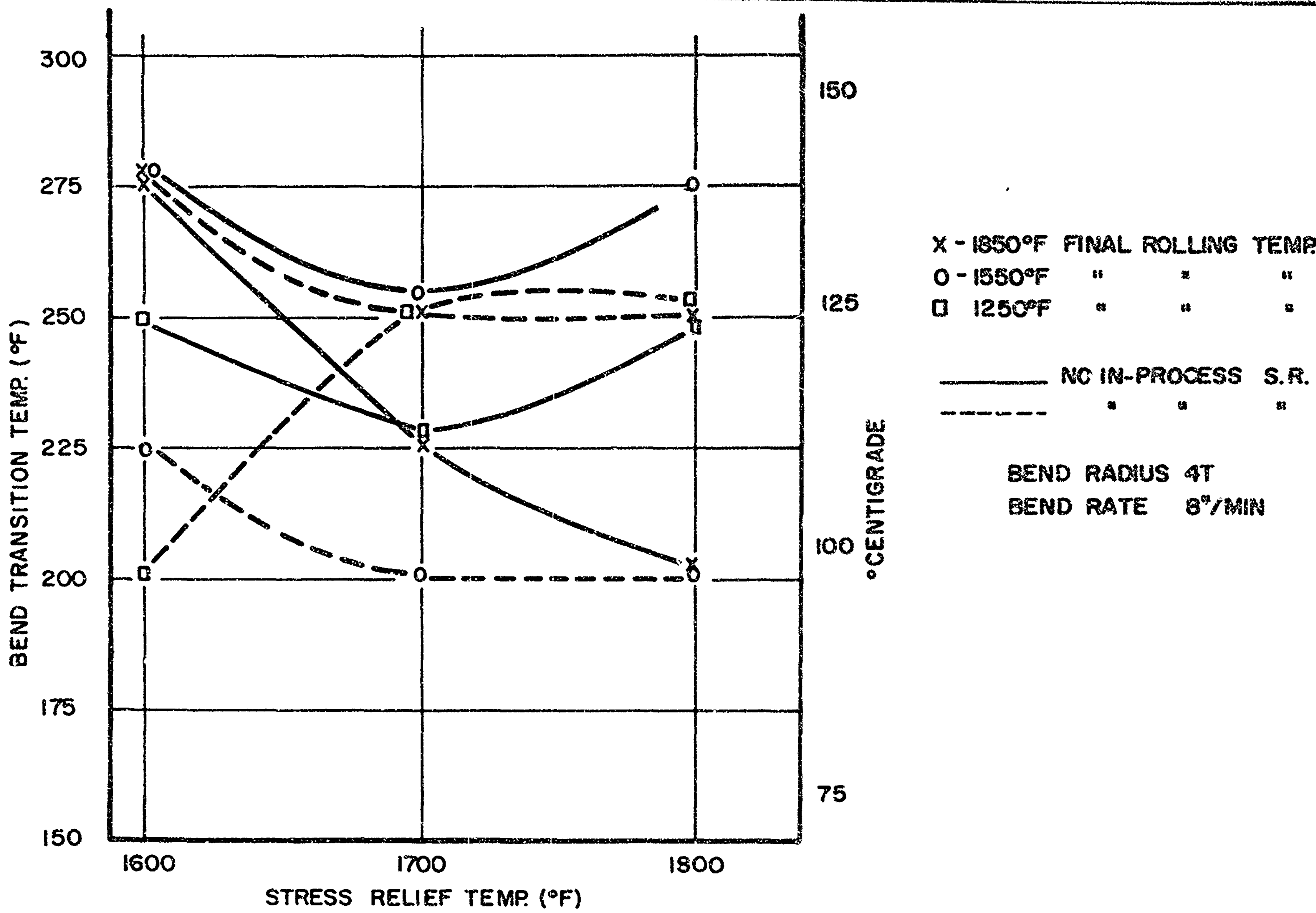


FIGURE 69

TRANSVERSE DUCTILE BRITTLE BEND TRANSITION OF .040" SHEET

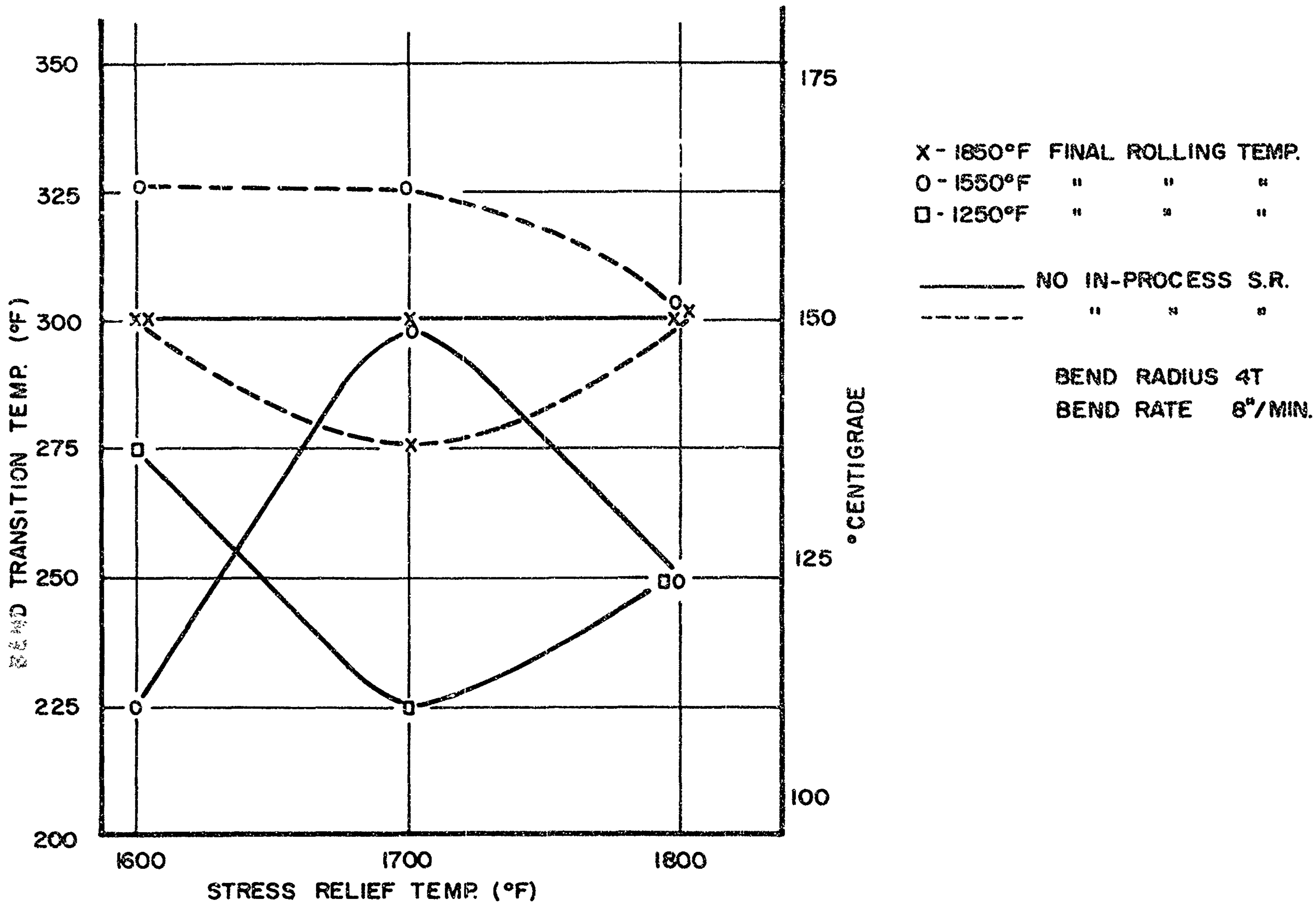


FIGURE 70

TRANSVERSE DUCTILE BRITTLE BEND TRANSITION OF .020" SHEET

delamination on cutting that sound samples could not be prepared. The relatively higher transition temperatures shown for the .020" material again point out the delamination problem. The two sheets having the lowest transition temperature both were rolled without an in-process anneal. Again, as occurred in both .040" and .060", the 1250° and 1550°F rolling temperatures were better than 1850°F. The lowest transition temperature for this material was 225°F.

The longitudinal bend transition data for sheets which displayed good results in the transverse direction are plotted in Figures 71 and 72. The transverse data are also plotted for comparison. Figures 68, 69, and 70 are identified as transverse properties; however, these data, in addition to micro-structural evaluation discussed later, show that although the transverse and longitudinal samples were cut with reference to the last rolling direction, the degree of cross rolling was not sufficient to erase the directionality established in the first rolling direction. The bend transition data, therefore, shows better properties in the transverse direction which is opposite to what would be expected. No attempt was made to run longitudinal tests on the .020" sheet because of the severe lamination problem.

Figure 71 contains a plot of the data for .060" sheet. It is shown that a compromise situation exists; i.e. if the sheet is anisotropic, one direction has an extremely low transition temperature and the other direction on this same material is relatively high. If the sheet is close to isotropic conditions, the difference between the longitudinal and transverse properties is very little. However, they are at a mid-point between the best and poorest conditions of the highly directional sheet.

In Figure 72, the same situation is shown to exist for .040" material as previously discussed for the .060" sheet. A further comparison of bend data can be made by

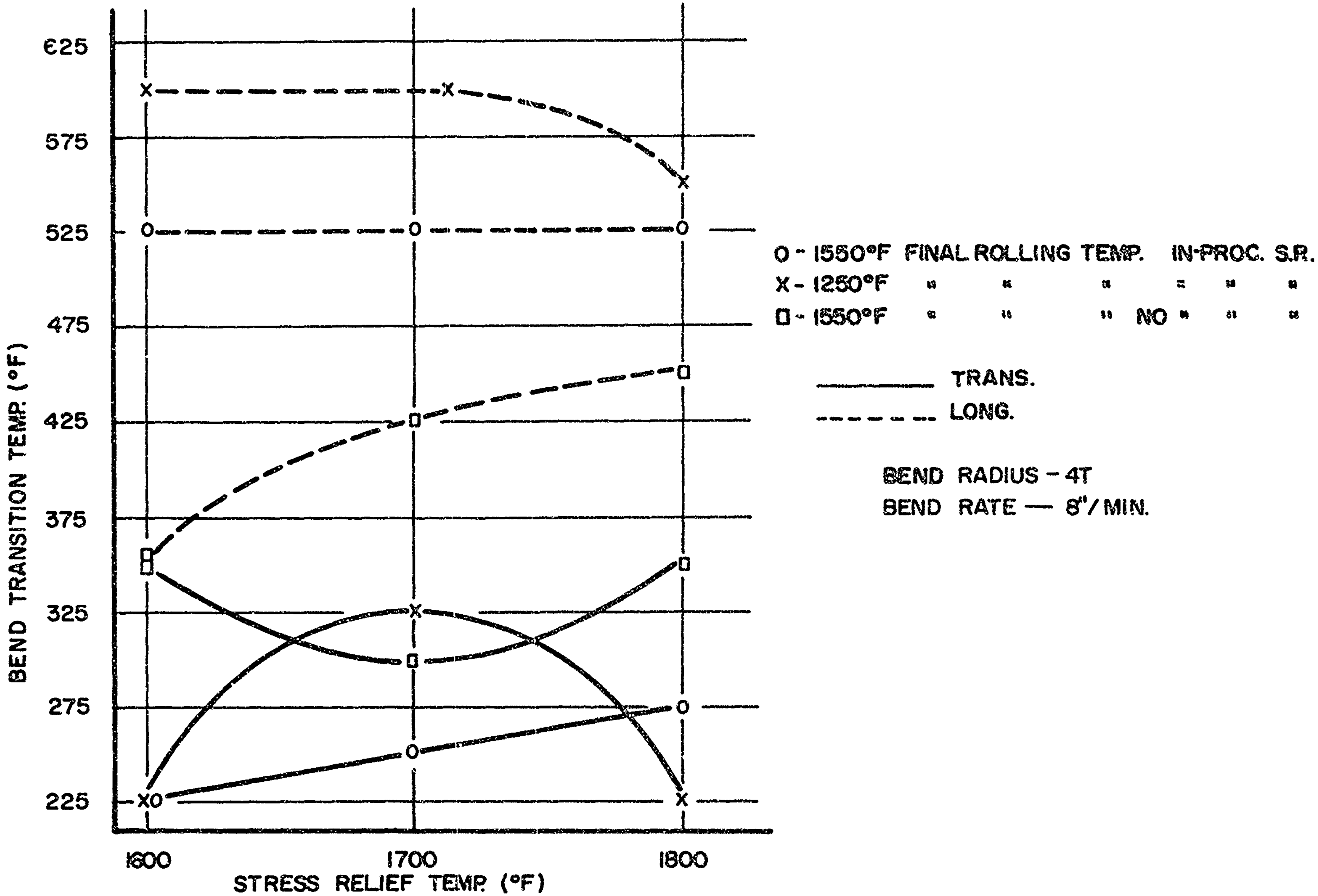


FIGURE 71

LONGITUDINAL & TRANSVERSE BEND TRANSITION TEMPERATURES OF THREE SELECTED .060" SHEETS

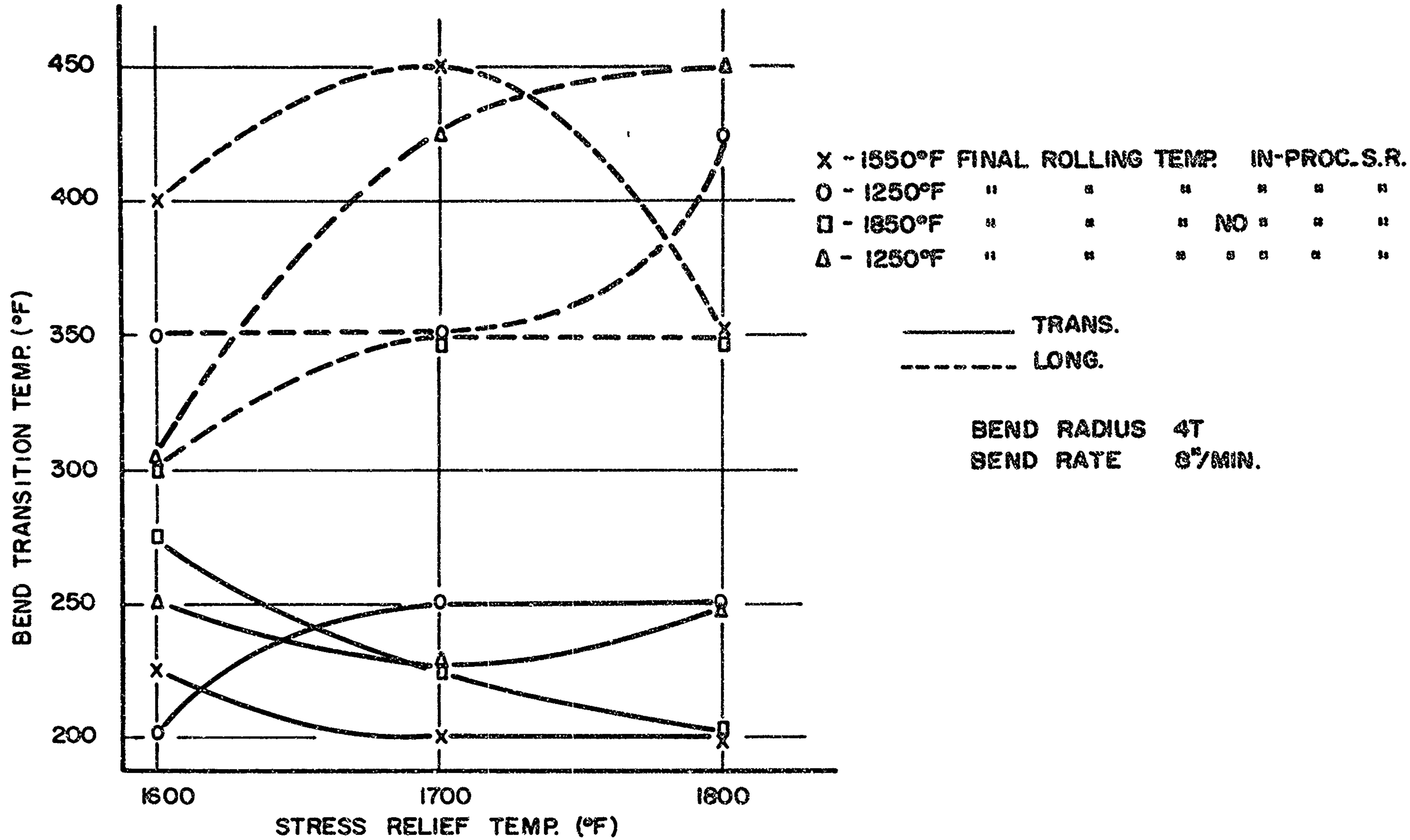


FIGURE 72

LONGITUDINAL & TRANSVERSE BEND TRANSITION TEMPERATURES OF FOUR SELECTED .040" SHEETS

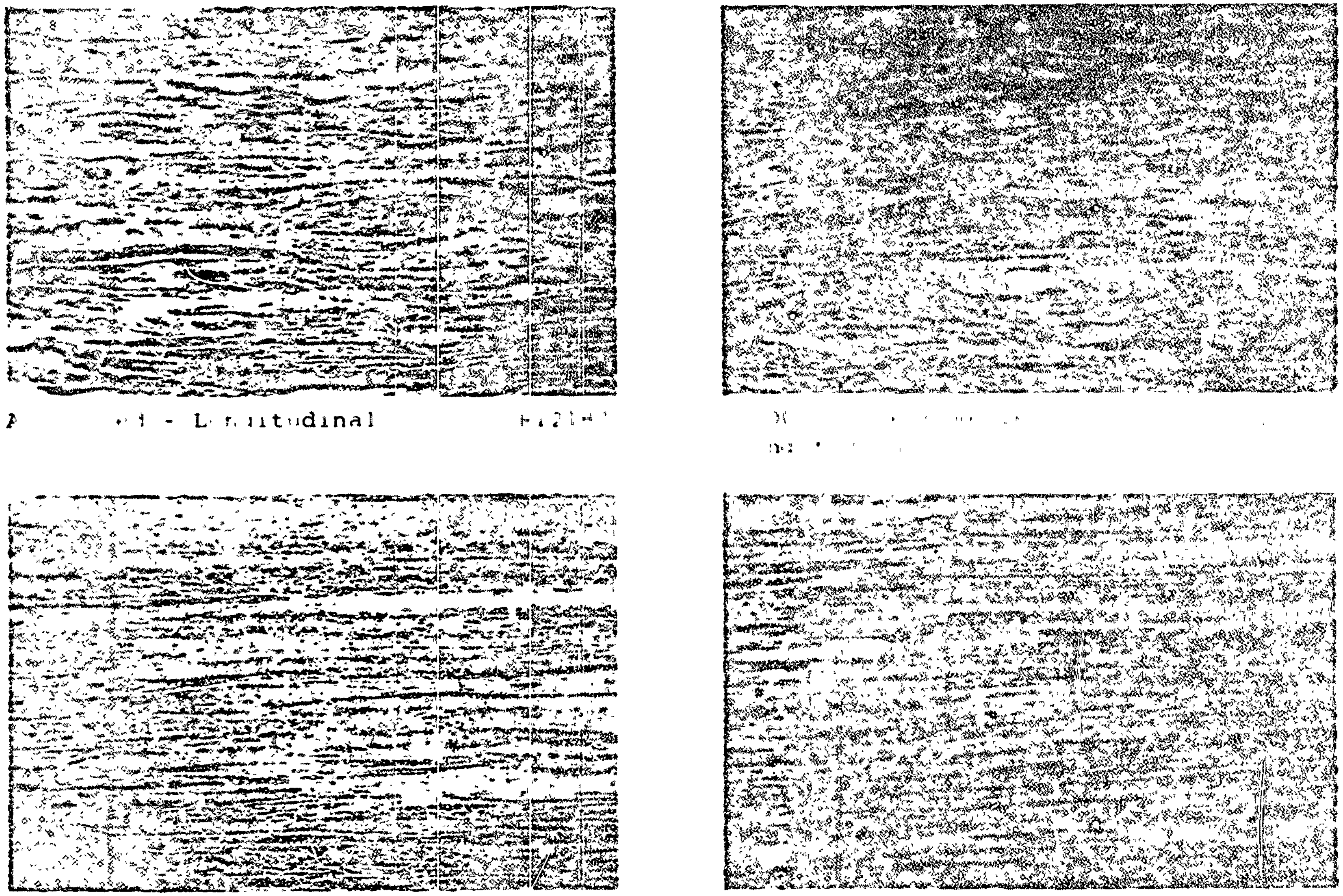
referring to the initial rolling studies previously covered. In this study, .040" gauge sheet was produced exclusively and no cross rolling was incorporated into the process schedule. By selecting four sheets from each practice, it is shown that cross rolling reduces the transverse bend transition temperature from an average 388° to 325°F. The minimum longitudinal transition temperature was 200°F in both cases; however, for cross rolled material, three out of four were at 200°F and in straight rolling, only one out of four was at this temperature. It is concluded then that cross rolling improves transverse properties and also maintains or improves longitudinal properties.

(7) Micro Examination

Samples from all sheets in the as-rolled 1600°, 1700°, and 1800°F stress relieved condition were examined metallographically. For each gauge, there was very little, if any, observable difference in the structures due to the various rolling practices. Typical structures of each gauge are shown in Figures 73, 74, and 75.

In reviewing these figures, it must be remembered that the directions indicated, i.e. longitudinal and transverse, are in relation to the final rolling direction. The typical wavy structure shown for the longitudinal direction is similar to that shown previously for the transverse structure in straight rolled material. It was concluded that the degree of cross rolling was not sufficient to erase or reverse the directionality established in the initial rolling direction from a microstructural aspect.

In Figure 75, representing .020" material, the longitudinal and transverse structures are shown to be almost identical. It is suggested that the cross rolling ratio of 1:1 used on this material should produce close to isotropic conditions.



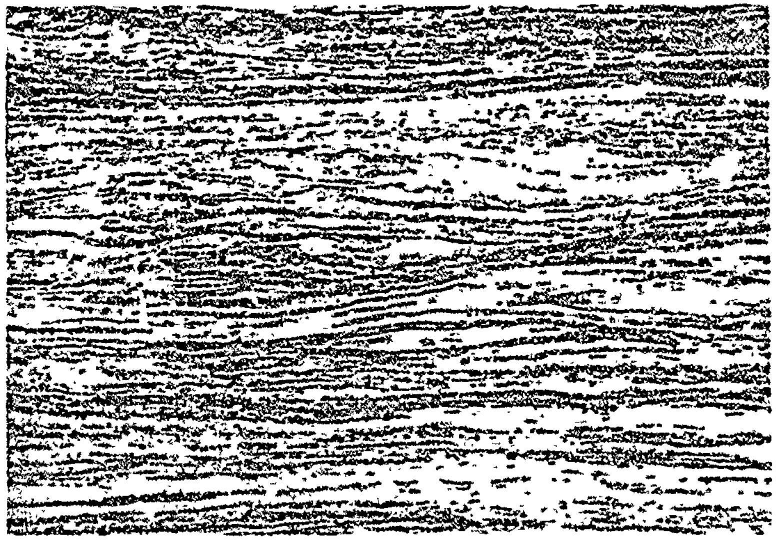
A[illegible] - L[illegible]itudinal

As-R[illegible]d - Transverse

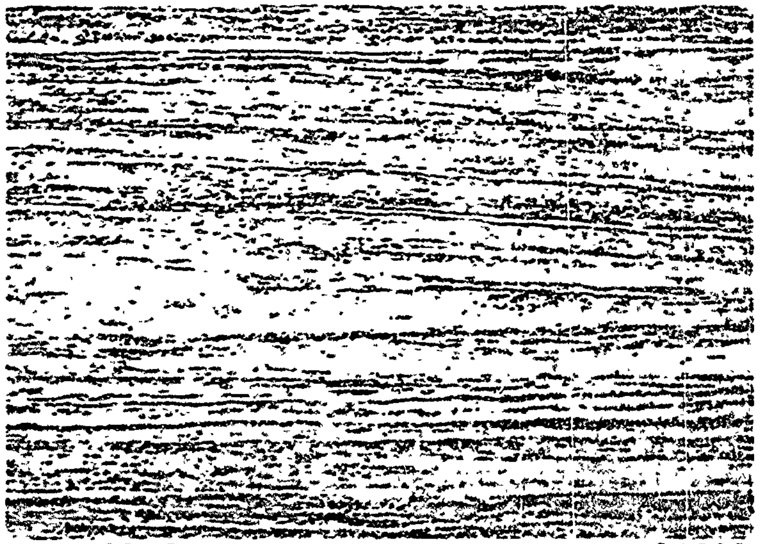
Figure 7:

Typical M[illegible]structures
Mag[illegible]ication

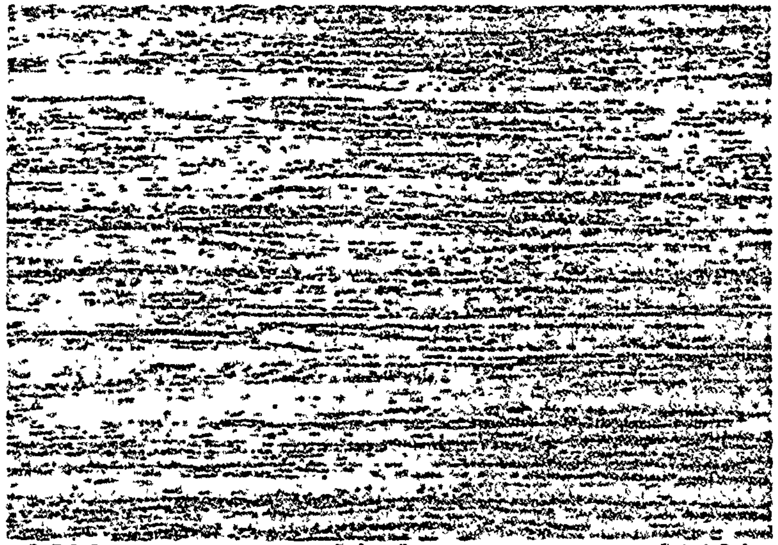
As-Rolled - Longitudinal R12199



1700°F Stress Relief R12203
Longitudinal



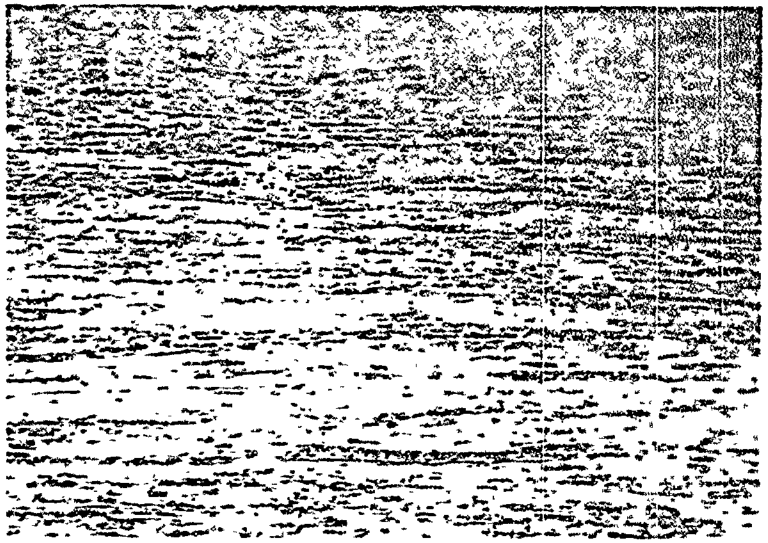
As-Rolled - Transverse R12200



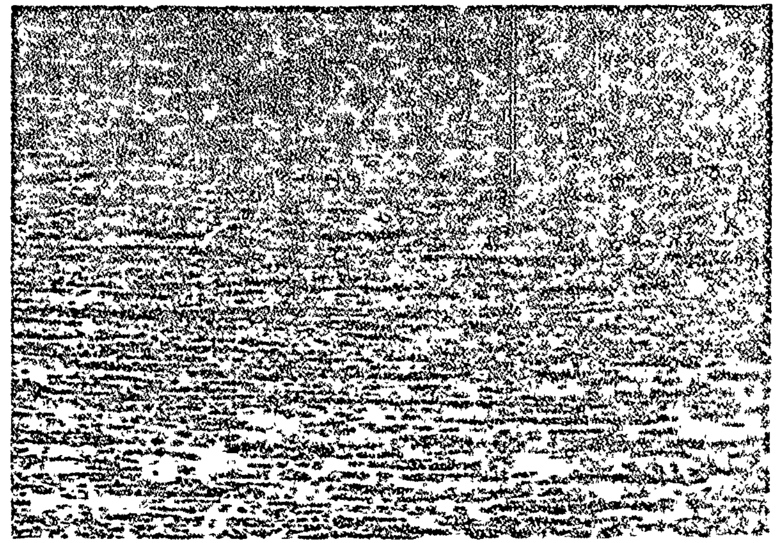
1700°F Stress Relief R12204
Transverse

Figure 74

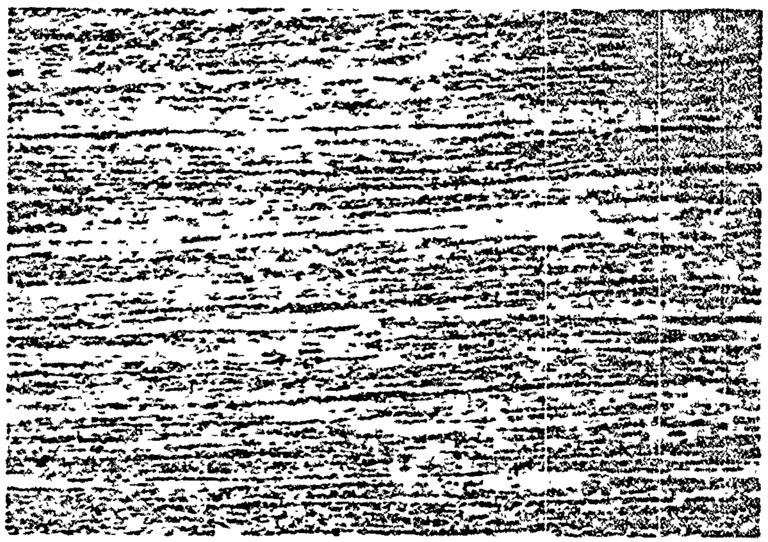
Typical Microstructures of .040" Sheet
Magnification 200X



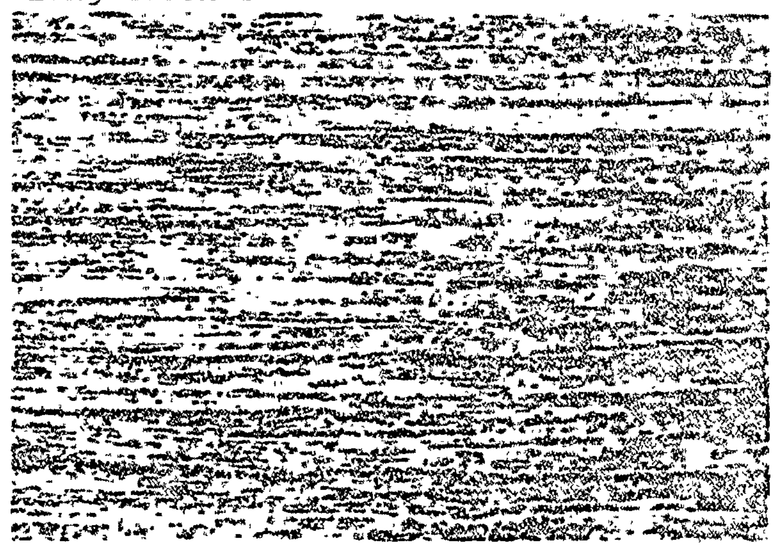
As-Rolled - Longitudinal R1219



1700°F Stress Relief R
Longitudinal



As-Rolled - Transverse R121



1700°F Stress Relief R
Transverse

Figure 75

Typical Microstructures of .020" Sheet
Magnification 200X

d. Analysis of Data

It appears from the tensile as well as bend and micro data that the high degree of work in the .020" material resulted in a high susceptibility to delamination upon cutting or shearing.

An overall analysis of the tensile data and, principally the elongation values, suggests the need for an in-process anneal. This is most clearly shown on the .060" material so that it is further concluded that the reduction after the in-process anneal should not exceed 88%, which corresponds to the process utilized for the .060" material.

From the lamination problems associated with the .020" material, it is concluded that the amount of cold work from the last recrystallization anneal was also too severe and a recrystallization anneal at some intermediate gauge will be required when producing this gauge sheet. It is concluded that the total work in the 0.040" and 0.060" sheet was close to optimum. The total reduction from the last recrystallization anneal should, therefore, not exceed 96% with 92% to 94% recommended.

It has been shown that of the three final rolling temperatures utilized (1850°, 1500°, and 1250°F), the two lower temperatures consistently resulted in better tensile elongation, ultimate tensile strength, and ductile-brittle bend transition.

Cross rolling at ratios of 2:1, 1.5:1, and 1:1 were utilized. The first two of these three did not permit sufficient work in the final rolling direction to produce isotropic properties. Unfortunately, the third ratio, 1:1 could not be completely evaluated due to the severe lamination problems in the .020" gauge material. Microstructural evaluation of this material did show that the longitudinal and transverse structures were quite

similar. It is concluded that a ratio approaching 1:1 would be desirable. This also agrees with the conclusion drawn by Pulsifer on powder metallurgy tungsten.

In selecting a final annealing temperature, a compromise situation again exists. It was shown that the strength drops off rapidly at increasing annealing temperatures above 1600°F (refer to Figure 63). This is more severe for the .060" sheet than the .040" and .020" (refer to Figures 64 and 65). With increasing annealing temperatures, the elongation values were, in general, increasing rapidly. The bend transition temperature was also shown to be effected by these low annealing temperatures. For tungsten, it is suggested that low temperature ductility should not be sacrificed for higher strength so that the final annealing temperature should be that which produces the highest tensile elongation and lowest bend transition temperature. It appears that the one hour 1700°F anneal best suits this prerequisite.

2. Scale-Up to 24" x 24" Sheet From 4" Diameter Extrusion Billet

a. Process Schedules for 24" x 24" Sheet

From the data presented, the rolling practice for producing the final sheets in this phase was established. One deviation from the selected conditions was the cross rolling ratio. It was established that a ratio approaching 1:1 would be desirable, but due to the configuration of the available sheet bar, it was impossible to produce 24" x 24" sheet having a 1:1 cross rolling ratio.

The process schedules established for each final gauge sheet were different and, therefore, are presented individually as follows:

Process Schedule for .060" Sheet

Starting Material:	Nominal 1" x 3" x 17" Sheet Bar
Condition:	Annealed 1 Hour at 2800°F
Roll To:	.400" x Nominal 7.5" x 17" at 2300°F
Stress Relieve:	One Hour at 2200°F
Roll To:	.100" x Nominal 27" x 17" at 2300°F
Condition	
Cross Roll To:	.060" x Nominal 25" x 27" at 1550°F
Stress Relieve:	One Hour at 1700°F
Crop To:	24" x 24"
Roller Level	
Descale	
Inspect	

Reduction After Recrystallization Anneal - 94%

Reduction After Stress Relief Anneal - 85%

Cross Rolling Ratio - 2.25:1

Process Schedule for .040" Sheet

Starting Material:	Nominal 1" x 3" x 11" Sheet Bar
Condition:	Annealed 1 Hour at 2800°F
Roll To:	.500" x Nominal 6" x 11" at 2300°F
Recrystallize:	One Hour at 2600°F
Roll To:	.250" x Nominal 12" x 11" at 2300°F
Stress Relieve:	One Hour at 2150°F
Roll To:	.100" x Nominal 27" x 11" at 2300°F
Condition	
Cross Roll To:	.040" x Nominal 25" x 27" at 1550°F
Stress Relieve:	One Hour at 1700°F
Crop To:	24" x 24"
Roller Level	
Descale	
Inspect	

Reduction After Recrystallization Anneal - 92%

Reduction After Stress Relief Anneal - 84%

Cross Rolling Ratio - 1.35:1

<u>Process Schedule for .020" Sheet</u>

Starting Material:	Nominal 1" x 3" x 7" Sheet Bar
Condition:	Annealed 1 Hour at 2800°F
Roll To:	.250" Nominal 12" x 7" at 2300°F
Recrystallize:	One Hour at 2600°F
Roll To:	.100" x Nominal 27" x 7" at 2300°F
Stress Relieve:	One Hour at 2100°F
Condition	
Cross Roll To:	.020" x Nominal 25" x 27" at 1550°F
Stress Relieve:	One Hour at 1700°F
Roller Level	
Descale	
Inspect	

Reduction After Recrystallization Anneal - 92%

Reduction After Stress Relief Anneal - 80%

Cross Rolling Ratio - .75:1

b. <u>Rolling Evaluation of 24" x 24" Sheet</u>

Using the process schedules outlined above, three sheet bars were rolled to produce the required .060", .040", and .020" sheet. The .060" sheet was rolled satisfactorily and after stress relieving, roller leveling and descaling, it was inspected for gauge tolerance and flatness. The results of this inspection were as follows:

<u>Flatness</u>

The flatness was measured using MAB recommendations. The MAB tolerance is 4%. The maximum deviation on this sheet was 2%.

Gauge Tolerance

The MAB tolerance on thickness is one-half of AMS2242. For .060", this is ±.003". Actual gauge measurements were as follows:

.058	.059	.058	.059	.060
.062	.062	.061	.061	
Rolling Direction ————>				
.061	.062	.062	.062	
.061	.061	.061	.062	.061

The maximum deviation from .060" was, therefore, +.002 and -.002.

The .040" sheet was inadvertently broke during rolling to final gauge due to an excessive time delay from furnace to mill caused by problems in aligning the sheet for rolling.

The .020" sheet was rolled to final size utilizing steel cover plates from .100". After rolling, the cover plates were removed and it was observed that the piece had folded or over-lapped in a plane parallel to the rolling direction in the center of the sheet on the trailing 10". This problem was attributed to improper roll crown and also to the fact that only one sheet was being rolled at the time. Multiple sheets would be used when more than one sheet is being produced. The sheet was subsequently evaluated for flatness and gauge tolerance with the following results:

Flatness

This piece was not roller leveled because of the fold on the trailing end. The as-rolled flatness was 3.5%.

Gauge Tolerance

The MAB tolerance for .020" gauge is ±.0015". At three points along each side, the actual gauge was as follows:

.0215	.0210	.0195
	Rolling Direction →	
.0195	.0207	.0200

The actual deviation was, therefore, +.0015 and -.0005.

D. Scale-Up to 36" x 36" Sheet From 6" Diameter Extrusion Billet

1. Sheet Bar Application

The material available for rolling to 36" x 36" sheet consisted of four mults of press forged sheet bar and three mults of direct extruded sheet bar produced from the evaluation of 6" diameter extrusion billet. Initially, for this investigation, it was planned to produce four sheets of each gauge to determine the process uniformity from sheet to sheet. Because of problems developed during rolling, some deviations from the above plans were required. In the first rolling series, two extruded sheet bars, mults 1148-1 and 1148-2 and two press forged sheet bars, 1167-1 and 1167-2, were applied to produce only the .060" sheets since these would be the easiest to produce and give experience in rolling the wide width.

2. Process Schedule for 36" x 36" Sheet

The most promising processing schedule for rolling 36" x 36" sheet in gauges of .060", .040", and .020" as determined by the initial rolling studies, is given in the following tables:

TABLE XXX

TENTATIVE ROLLING SCHEDULE FOR .060" SHEET

1. Roll from sheet bar to 1" thick at 2300°F furnace temperature.
2. Recrystallize one hour at 2700°F.
3. Roll to .400" thick at 2300°F furnace temperature.
4. Stress relieve one hour at 2000°F.
5. Roll to 38" long x width x gauge at 2300°F (nominal 38" x 20" x .120").
6. Cross roll to .060" x 38" wide x length at 1400°F (nominal 40" long).
7. Crop to 36" x 36".
8. Roller level at 1400°F furnace temperature.
9. Descale.
10. Stress relief at 1700°F.
11. Inspect and evaluate.

TABLE XXXI

TENTATIVE ROLLING SCHEDULE FOR .040" SHEET

1. Roll from sheet bar to .600" thick at 2300°F furnace temperature.
2. Recrystallize one hour at 2600°F.
3. Roll to .300" thick at 2300°F furnace temperature.
4. Stress relieve one hour at 2000°F.
5. Roll to 38" long x width x gauge at 2300°F (nominal 38" x 24" x .150").
6. Cut into two pieces nominal 38" x 12" x .150".
7. Cross roll to .040" x 38" wide x length at 1400°F (nominal 38").
8. Shear to maximum size.
9. Roller level at 1400°F furnace temperature.
10. Descale.
11. Stress relieve.
12. Inspect and evaluate.

TABLE XXXII

TENTATIVE ROLLING SCHEDULE FOR .020" SHEET

1. Roll from sheet bar to .275" thick at 2300°F furnace temperature.
2. Recrystallize one hour at 2500°F.
3. Roll to .125" thick at 2100°F.
4. Stress relieve one hour at 2000°F.
5. Cross roll to .060" x 38" wide at 1400°F.
6. Cut in half (.060" x 38" x 14" nominal).
7. Roll to .020" at 1400°F (nominal .020" x 38" x 40").
8. Shear to maximum size.
9. Roller level at 1400°F furnace temperature.
10. Descale.
11. Stress relieve at 1700°F.
12. Inspect and evaluate.

3. Rolling Evaluation of 36" x 36" Sheet

a. .060" Gauge Sheet Product

The four originally applied sheet bars were rolled to an intermediate gauge of 1" using a 2300°F furnace temperature. One pass per reheat was used for this initial rolling step.

The initial breakdown of the four sheet bars resulted in no visual cracks. Rolling to final gauge required three additional steps, as outlined in Table XXX. The material was next rolled from 1" thick to .400" at a temperature of 2300°F. The first piece cracked severely on the second pass. This was attributed to excessive reductions per pass and by decreasing the reduction per pass on the remaining three pieces, there were no further problems. At this point, the three remaining pieces were stress relieved and conditioned for further rolling. Material salvaged from the cracked piece was reapplied to .020" gauge.

The third rolling operation was then undertaken to roll the material to .120". In this rolling operation, sheet 1148-2 was initially a heavier starting gauge and one pass was required to get it equivalent to the remaining two pieces. After the one pass was rolled, the mill settings were the same for all pieces down to a point where a 38" length for cross rolling was acquired. After this rolling operation, the sheets were sheared for cross rolling. During shearing, sheet 1148-2 cracked, thus preventing production of a full sized .060" x 36" x 36" sheet. The actual sizes of the three remaining sheets after shearing and conditioning were as follows:

1148-2	.117" x 38" long x 13-1/2" wide
1167-1	.112" x 38" long x 20-1/2" wide
1167-2	.106" x 38" long x 19" wide

Since the first piece (sheet 1148-2) could not be rolled to a full size at .060" gauge, the schedule was changed to permit rolling to .040" which would produce a full 36" x 36" sheet.

For the fourth rolling operation, cover plates of AISI 1095 steel were used to facilitate reduction to the desired gauge. Two sheets, nominal .125" thick, were cut to match each of the three tungsten sheets. During this rolling operation, the gauge was not measured until the sheets were close to the desired final thickness. Reduction in this case was determined by the increasing length of the total pack. The two pieces being rolled to .060" gauge rolled satisfactorily although some edge cracking did occur. This edge cracking had previously been explained under the initial rolling evaluation as being caused by misalignment of the cover sheets with respect to the tungsten thus causing uneven reduction and stresses on the edge sections. The nominal as-rolled size of these pieces was 38" wide x 40" long. The piece reapplied

to .040" developed a longitudinal crack in the center of the trailing end early in the rolling sequence. Although this crack did not propagate, it did elongate proportional to the reduction. At .040" gauge, the piece was a nominal 38" wide x 41" long. However, the crack permitted only a 26" length of sound material.

After rolling, all three pieces were sheared to maximum size. During the shearing operation, a crack developed along the side of piece 1167-1 which required additional shearing to 33-1/2" wide x 37" long. On piece 1167-2 a crack developed on the end which required shearing to a length of 32-1/2" x 36-3/4" wide. The three sheets were subsequently roller leveled utilizing a 1400°F furnace temperature. During the flattening operation, a crack developed on piece 1148-2 which required shearing to 35" wide. After the flattening operation, the sheets were descaled in molten caustic. Figures 76 and 77 show the sheets .060" and .040" respectively. Stains are prevalent from the descaling operation; however, these are readily removed by scrubbing.

(1) Inspection

The flatness and gauge tolerance are summarized in Table XXXIII. The goals on gauge control were one-half of AMS 2242 tolerances which are ±.002 and ±.003 for .040" and .060" sheet respectively. As shown in the table, the .040" sheet was out of specification by 0.001" on the high side with the minimum value being 0.0400". Since the total variation was 0.003", the sheet could readily be pickled to uniformly remove approximately .0015". This should then give a .olerance of 0 040", ±.0015". Both .060" sheets were within the desired gauge tolerance. The degree of bow or out-of-flat as determined using MAB recommended practices are less than the maximum 4%.

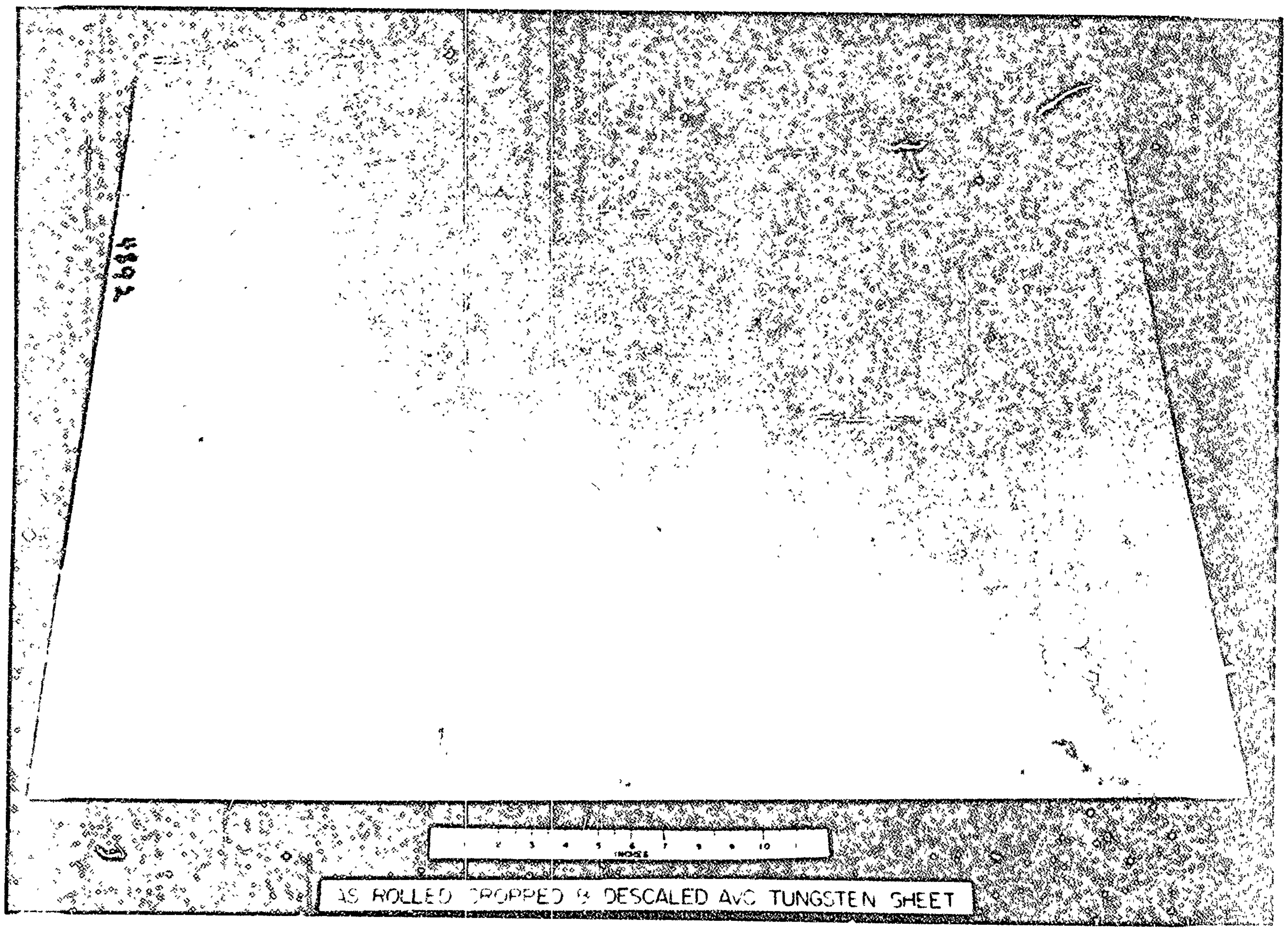


Figure 76

Sheet .060" x 37" Wide x 33" Long

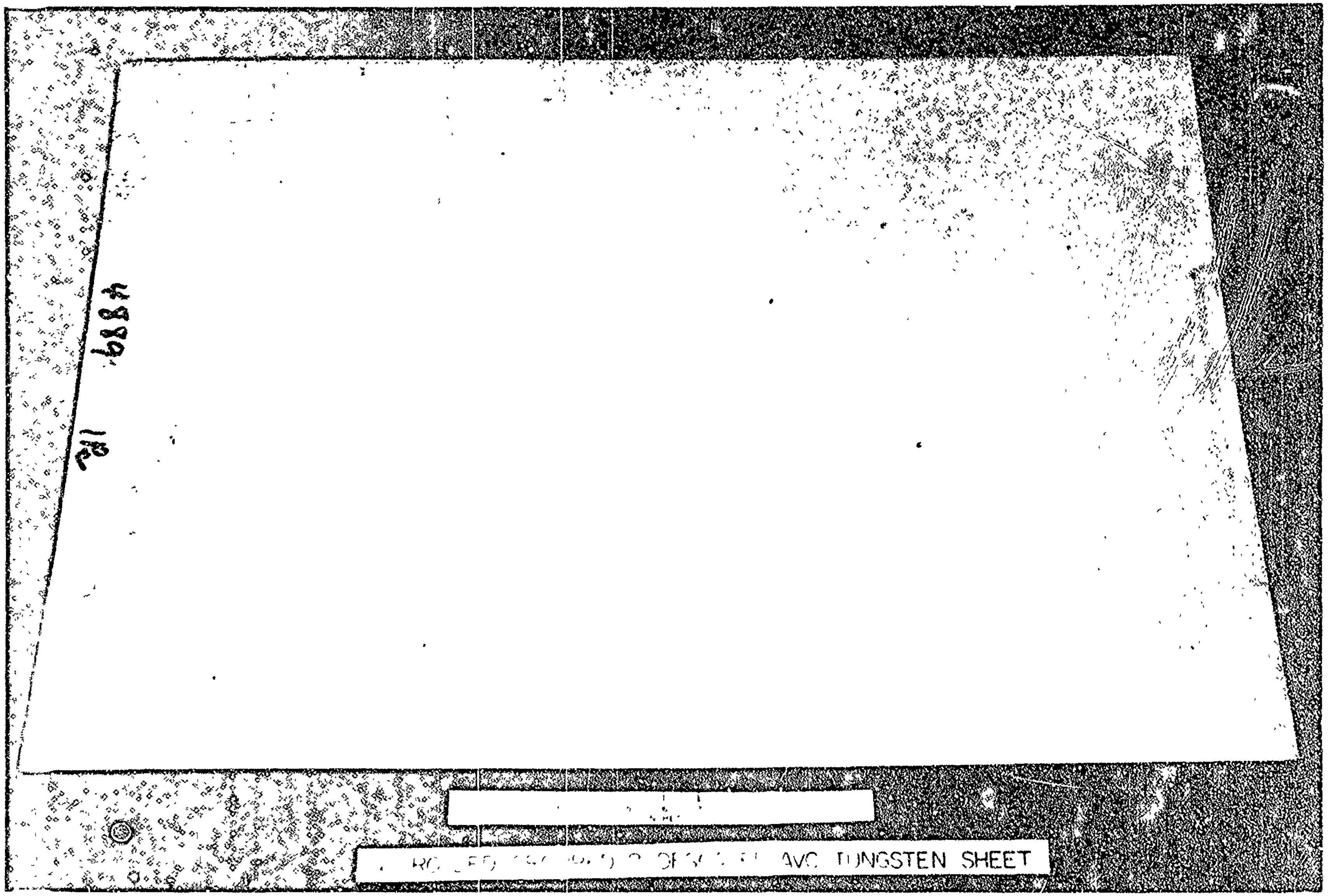


Figure 77

Sheet .040" x 36" Wide x 26" Long

TABLE XXXIII

GAUGE AND FLATNESS SURVEY

	1148-2	1167-1	1167-2
Gauge 1" From Edge	.040 x 36w x 26	.060 x 33-1/2w x 37	.060 x 36-3/4w x 32-1/2
Average	.0416	.0595	.0596
Maximum	.0427	.0620	.0612
Minimum	.0400	.0577	.0577
Tolerance			
Desired Gauge	$.040^{+.0027}_{-.0000}$	$.060^{+.0020}_{-.0023}$	$.060^{+.0012}_{-.0023}$
Average	$.0416^{+.0011}_{-.0016}$	$.0595^{+.0025}_{-.0018}$	$.0589^{+.0023}_{-.0012}$
Gauge 3" From Edge			
Average	.0417	.0610	.0608
Maximum	.0430	.0627	.0617
Minimum	.0410	.0595	.0597
Tolerance			
Desired Gauge	$.040^{+.0030}_{-.0000}$	$.060^{+.0027}_{-.0005}$	$.060^{+.0017}_{-.0003}$
Average	$.0417^{+.0010}_{-.0007}$	$.0610^{+.0017}_{-.0015}$	$.0608^{+.0009}_{-.0011}$
% Out-Of-Flat	3.75	3.75	3.67

All Averages Are Based on a Minimum of 20 Readings
Divided Equally Along All Four Sides

Desired Tolerances:	Gauge (1/2 AMS2242)	$0.040\pm.002$	$0.060\pm.003$
	Flatness	Less Than 4%	

(2) Microscopic Examination

Samples from each sheet were annealed at 1700°F and prepared for micro examination. Figure 78 shows the representative longitudinal and transverse microstructures. There is no apparent difference between the sheets which can be observed from these microphotographs.

(3) Bend Transition

The 4T bend transition temperature was investigated using a constant 8" per minute ram speed. Samples for both tensile and bend studies were cut from both the left and right side of each sheet for determination of the uniformity within the sheets. Both longitudinal and transverse properties were investigated.

Several final annealing temperatures were investigated which required shearing the test samples out of the sheet in the as-rolled condition. This later proved to be a problem since severe delamination resulted from shearing.

The sequence of sample cutting and preparation was as follows:

.060" Samples

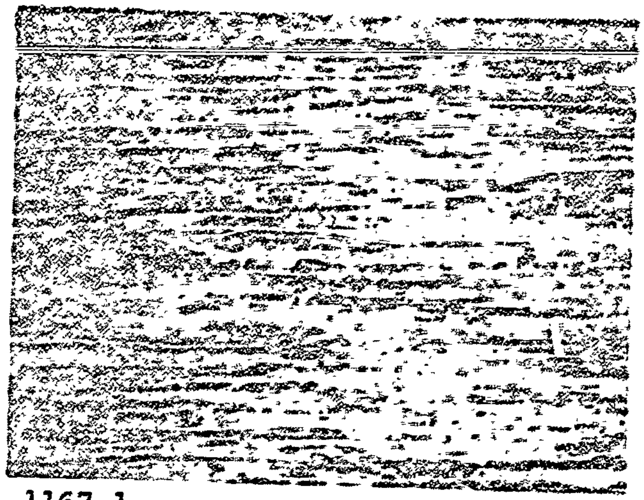
Shear 1.20" wide strips.

Abrasive cut strips into .750" wide samples (samples .750" wide x 1.20" long or 12-1/2T wide x 20T long).

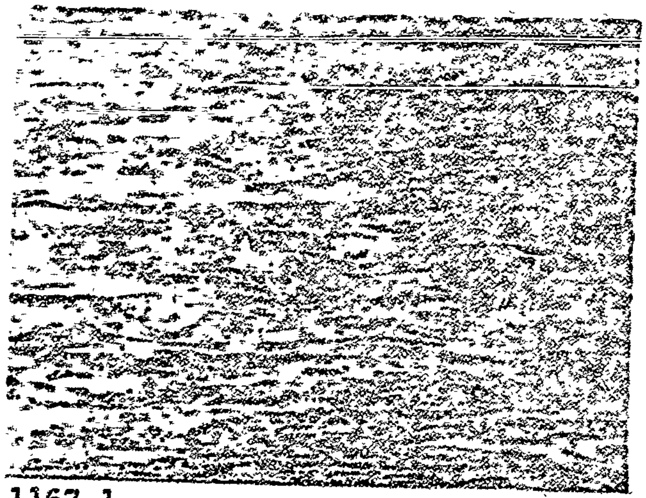
Polish edges to be bent (abrasive cut edges) through 150 grit paper.

Test.

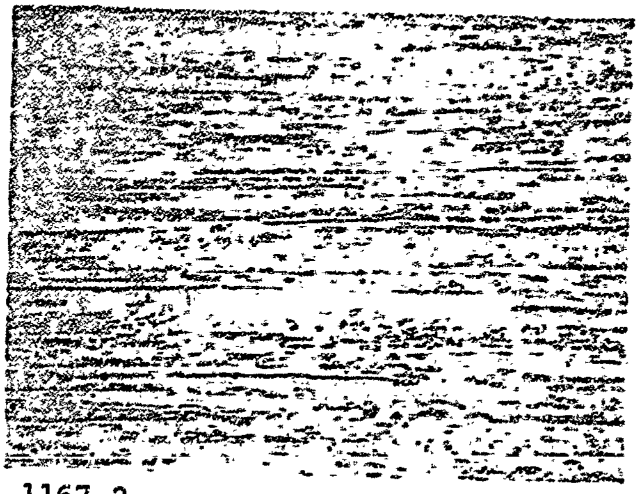
The .040" samples were prepared using the same procedure. The size was smaller, consistent with the nominal 12T width and 20T length.



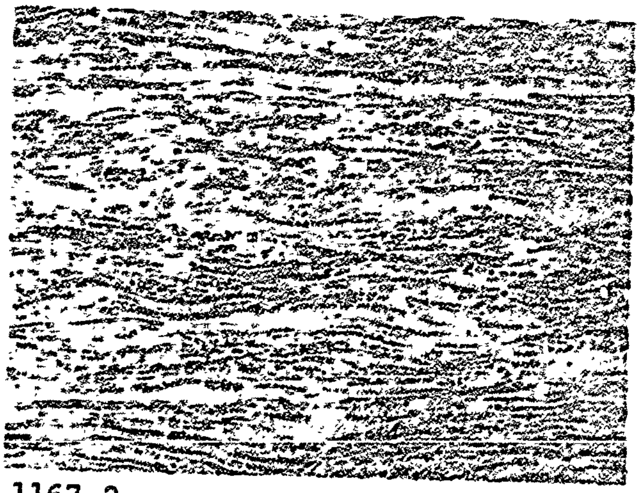
1167-1 R13060
.060" Transverse



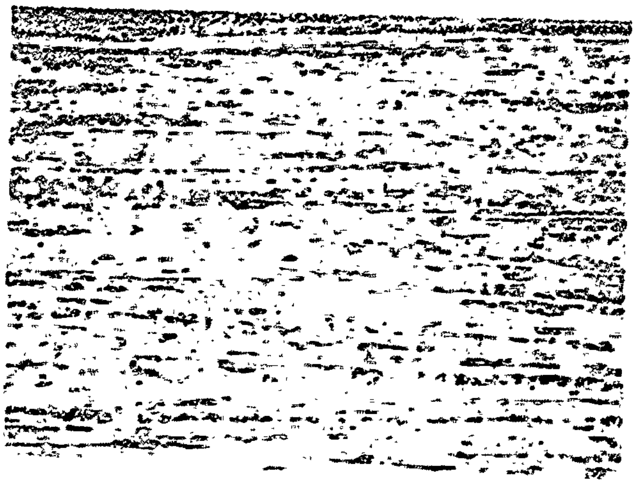
1167-1 R13057
.060" Longitudinal



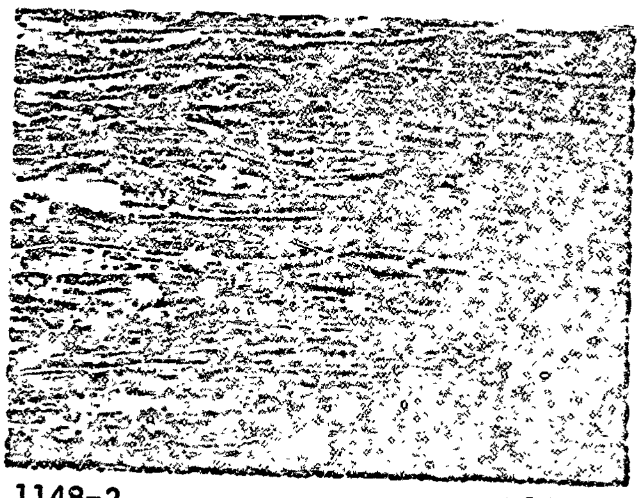
1167-2 R13061
.060" Transverse



1167-2 R13058
.060" Longitudinal



1148-2 R13062
.040" Transverse



1148-2 R13059
.040" Longitudinal

Figure 78

Typical Longitudinal and Transverse Microstructures
Magnification 200X

The edges across which the bending takes place were abrasive cut while the ends were sheared. Inspection of the samples using 10X magnification showed in many cases delamination starting at the sheared edge and extending up to 1/4" through the sample. This was not felt to be detrimental as they did not extend into the actual area of the bend. The die and ram were mounted in a universal testing machine using a resistance heated muffle furnace. A thermocouple was attached to the top face of the die adjacent to the specimen. Figure 79 is a plot of the bend transition data for the two sheets of .060" gauge. Considering only the temperatures at which a full bend can be made, it is shown that:

1. The variation in transverse values within one sheet from the left to the right side is 50°F.
2. The maximum variation in the transverse direction between the two sheets is 100°F.
3. The variation in longitudinal values within one sheet is 70°F.
4. The variation in the longitudinal direction between two sheets is 75°F.

(4) Tensile Properties

For all initial tensile testing samples having a 3/16" gauge width and 3/4" gauge length were utilized. Table XXXIV lists the partially completed data or investigation of the tensile transition. In reviewing this table, the first obvious point is that in this low temperature region, most of the samples broke at the pin hole. This problem did not exist on elevated temperature tests. As the test temperature decreases, the unit

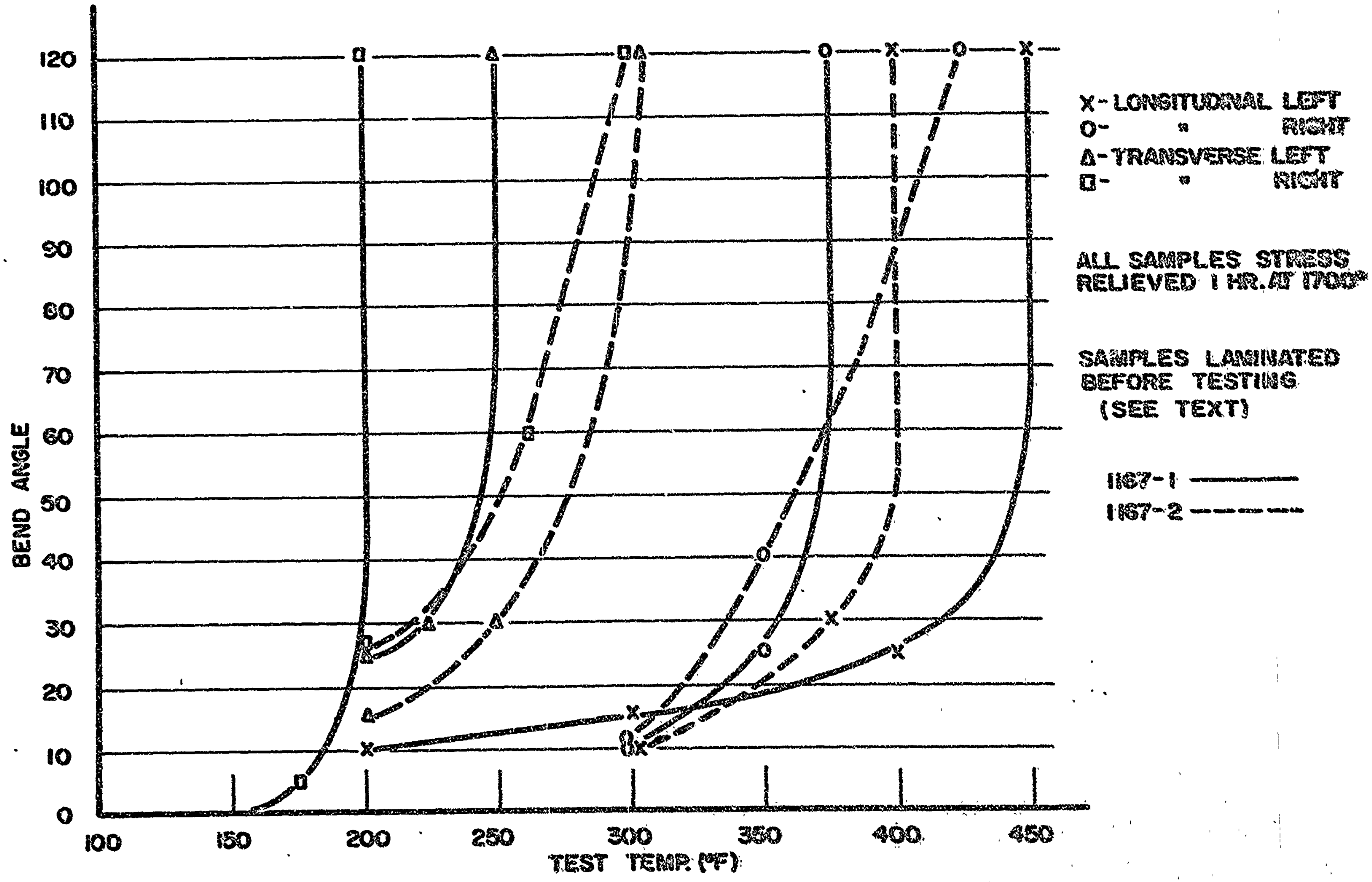


FIGURE 79

4T - BEND TRANSITION TEMPERATURES FOR .060" SHEET

TABLE XXXIV

TENSILE TRANSITION DATA

Sheet 1167-1

Test Direction	Final Anneal	Test Temperature °F	UTS $\times 10^3$	.2% YS $\times 10^3$	% Elongation	Comments
Longitudinal	1600°F	300	162.7	145.5	10.5	
Longitudinal	1600°F	250	145.2	--	--	Premature Break at Pin Hole
Longitudinal	1600°F	250	178.7	165.2	4.5	
Longitudinal	1600°F	200	107.4	--	--	Premature Break at Pin Hole
Longitudinal	1600°F	200	182.2	174.5	--	Premature Break at Pin Hole
Longitudinal	1600°F	225	139.9	--	--	Premature Break at Pin Hole
Transverse	1600°F	300	172.4	160.0	4.8	
Transverse	1600°F	250	164.2	--	--	Premature Break at Pin Hole
Transverse	1600°F	250	186.9	168.8	7.2	
Transverse	1600°F	200	132.9	--	--	Premature Break at Pin Hole
Transverse	1600°F	200	178.6	--	--	Premature Break at Pin Hole
Transverse	1600°F	225	146.8	--	--	Premature Break at Pin Hole
Longitudinal	1800°F	300	156.7	139.2	11.3	
Longitudinal	1800°F	250	170.1	154.7	3.5	
Longitudinal	1800°F	200	178.3	163.3	--	Premature Break at Pin Hole
Longitudinal	1800°F	200	179.8	161.9	3.7	
Longitudinal	1800°F	150	112.9	--	--	Premature Break at Pin Hole
Longitudinal	1800°F	175	112.1	--	--	Premature Break at Pin Hole
Transverse	1800°F	300	168.8	152.6	10.7	
Transverse	1800°F	250	152.0	--	--	Premature Break at Pin Hole
Transverse	1800°F	250	173.6	163.3	8.9	
Transverse	1800°F	200	189.9	176.5	--	Premature Break at Pin Hole
Transverse	1800°F	200	158.1	--	--	Premature Break at Pin Hole
Transverse	1800°F	225	161.2	--	--	Premature Break at Pin Hole

Test Parameters -- .005"/in/min to .6% Yield
.05"/in/min to Fracture

strength is increasing rapidly, i.e., at 900°F the nominal UTS is 120,000 psi while values in the range of 200° to 300°F are as high as 190,000 psi. The load required for the low temperature tests is sufficiently higher to cause the grip area (pin hole) to fracture prematurely. In addition, the notch sensitivity in the ductile-brittle region is much greater.

In continuing the tests up to 900°F, a very interesting phenomena occurred. In Figure 80, it is shown that the ultimate tensile strength and .2% yield strength decreased uniformly with increasing temperature; however, the elongation, starting in the ductile-brittle zone increased rapidly to a peak at 400°F and then decreased to approximately 600°F, where the rate of decrease becomes more gradual, reaching a low point at 900°F. This is in agreement with previously reported literature data; however, the results appear more pronounced in this work.

Based on MAB standards, extensive testing of all sheets was accomplished at 900°F. Duplicate samples from two positions within each sheet were tested in both the longitudinal and transverse directions. Table XXXV lists the results obtained for samples stress relieved at 1700°F. The sample position, i.e. left and right, refer to opposite sides of the sheets where samples were taken.

In reviewing the data in Table XXXV, the variation in properties can be summarized as follows:

Maximum Range on Duplicate Samples

	UTS	.2% Yield	% Elongation
	115,700-121,500	98,100-112,500	7.6-8.7
Mean	118,600 ±2,900	105,300 ±7,200	8.1 ±0.5

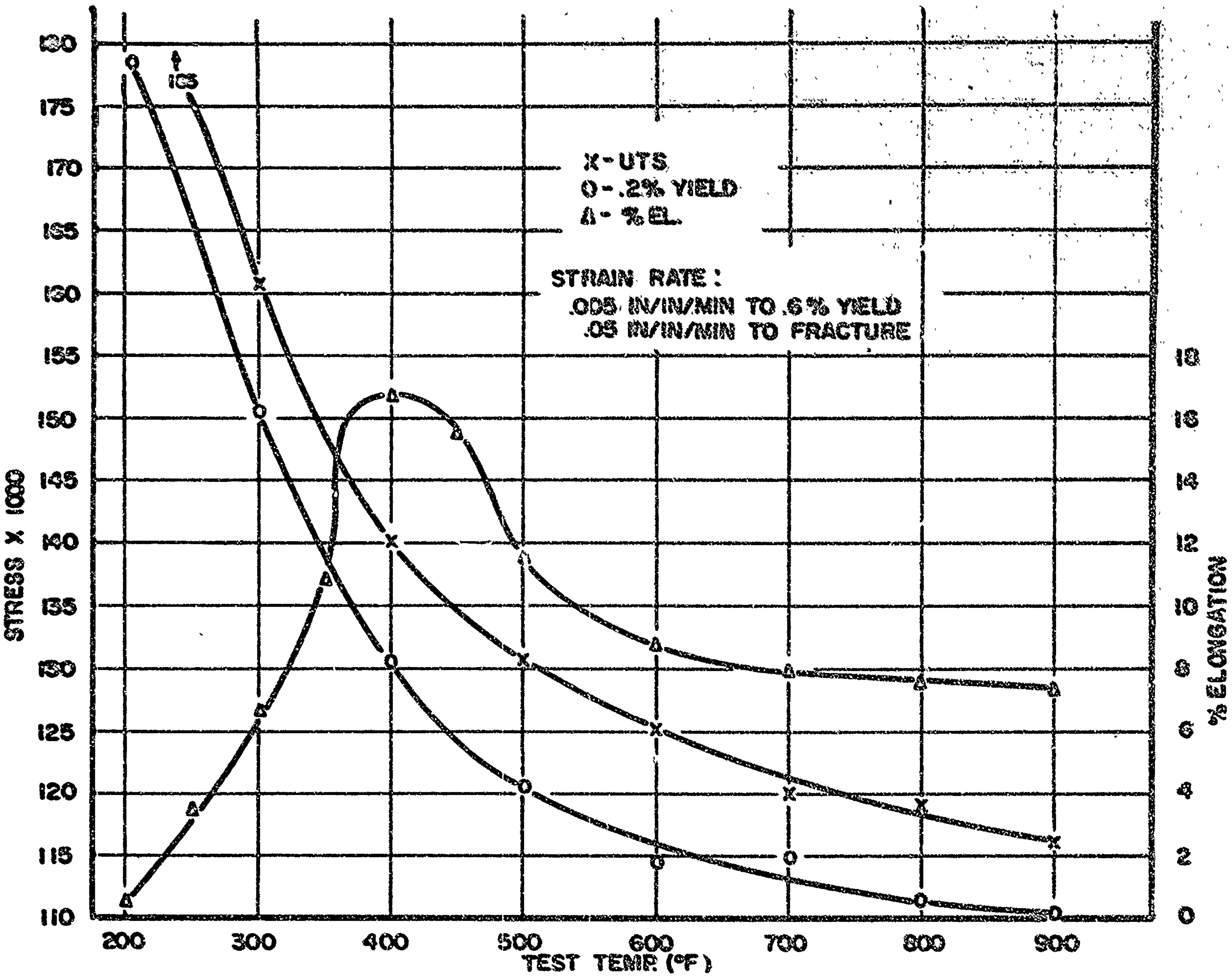


FIGURE 80

LOW TEMPERATURE TENSILE PROPERTIES (200-900°F)

TABLE XXXV

900°F TENSILE PROPERTIES

Final Stress Relief 1700°F

Sheet Number	Sample Position	Test Direction	UTS $\times 10^3$	.2% Yield $\times 10^3$	% Elongation
1148-2	Right	Transverse	123.1	107.1	4.7
	Right	Transverse	121.8	102.1	5.6
	Left	Transverse	121.8	106.9	6.0
	Left	Transverse	121.2	106.6	5.2
	Right	Longitudinal	115.6	105.7	5.9
	Right	Longitudinal	115.4	98.3	5.7
	Left	Longitudinal	115.7	98.1	6.3
	Left	Longitudinal	121.5	112.5	6.0
1167-1	Right	Transverse	115.5	108.7	7.3
	Right	Transverse	116.3	110.1	7.9
	Left	Transverse	113.7	105.1	8.1
	Left	Transverse	115.0	106.4	8.5
	Right	Longitudinal	108.6	93.9	7.6
	Right	Longitudinal	111.3	100.5	6.7
	Left	Longitudinal	110.1	104.4	8.0
	Left	Longitudinal	110.7	98.0	8.7
1167-2	Right	Transverse	111.2	100.7	8.3
	Right	Transverse	112.0	103.3	8.3
	Left	Transverse	119.9	110.1	8.3
	Left	Transverse	120.1	114.1	7.2
	Right	Longitudinal	118.5	108.9	7.9
	Right	Longitudinal	117.3	107.4	7.9
	Left	Longitudinal	110.0	103.8	7.6
	Left	Longitudinal	110.5	95.6	8.7

Maximum Range on Samples From One Sheet One Direction

	UTS	.2% Yield	% Elongation
	111,200-120,100	98,100-112,500	6.7-8.7
Mean	115,700 ±4,500	105,300 ±7,200	7.7 ±1

Maximum Range on All Samples From One Sheet (Longitudinal and Transverse)

	UTS	.2% Yield	% Elongation
	110,000-120,100	95,600-114,100	7.7-8.7
Mean	115,050 ±5,050	104,800 ±9,200	7.7 ±1

Maximum Range on All Samples From All Sheets

	UTS	.2% Yield	% Elongation
	108,600-123,100	93,000-114,100	4.7-8.7
Mean	115,850 ±7,250	104,000 ±10,000	6.7 ±2

The allowable room temperature variations established by MAB for all specimens are:

UTS - ±7% About the Mean

.2% Yield - ±10% About the Mean

The above data shows that the percent variation on all samples was:

UTS - ±6.27%

.2% Yield - ±9.72%

The average elongation for all samples is 7.2%. The average of the .040" samples is 5.68% and for the .060" only, 7.95%. The lower elongation in the .040" samples is attributed to the fact that this material was originally scheduled to .060" and, therefore, the intermediate anneals were such that at .040" the sheet contained more work than the .060" sheet.

As previously stated, the data in Table XXXV was for samples having a final stress relief of 1700°F. On one sheet (1167-2) additional samples were tested from the right side only after 1600° and 1800°F anneals. The results are listed in Table XXXVI.

TABLE XXXVI

900°F TENSILE PROPERTIES
FINAL STRESS RELIEF 1600° AND 1800°F
SHEET 1167-2 ONLY

Anneal	Direction	UTS x 10^3	.2% Yield x 10^3	% Elongation
1600	Transverse	124.2	119.5	6.5
1600	Transverse	123.0	115.2	7.3
1600	Longitudinal	107.7	104.1	5.9
1600	Longitudinal	117.6	109.8	7.5
1800	Transverse	113.7	102.4	8.3
1800	Transverse	114.2	105.6	6.9
1800	Longitudinal	109.8	97.7	7.2
1800	Longitudinal	107.4	101.8	7.7

The average values for the three anneals on this sheet were as follows:

Anneal	UTS x 10^3	.2% Yield x 10^3	% Elongation
1600	118.1	112.15	7.55
1700	114.8	105.1	8.10
1800	111.3	101.9	7.52

The UTS and .2% Yield decrease with increasing annealing temperature is as would be expected. The elongation values are shown to be the same for 1600° and 1800°F, but slightly higher for the 1700°F anneal.

Tensile properties were investigated in the temperature range of 900° to 2000°F. Figure 81 shows that a gradual decrease in strength occurs, the UTS decreasing from a

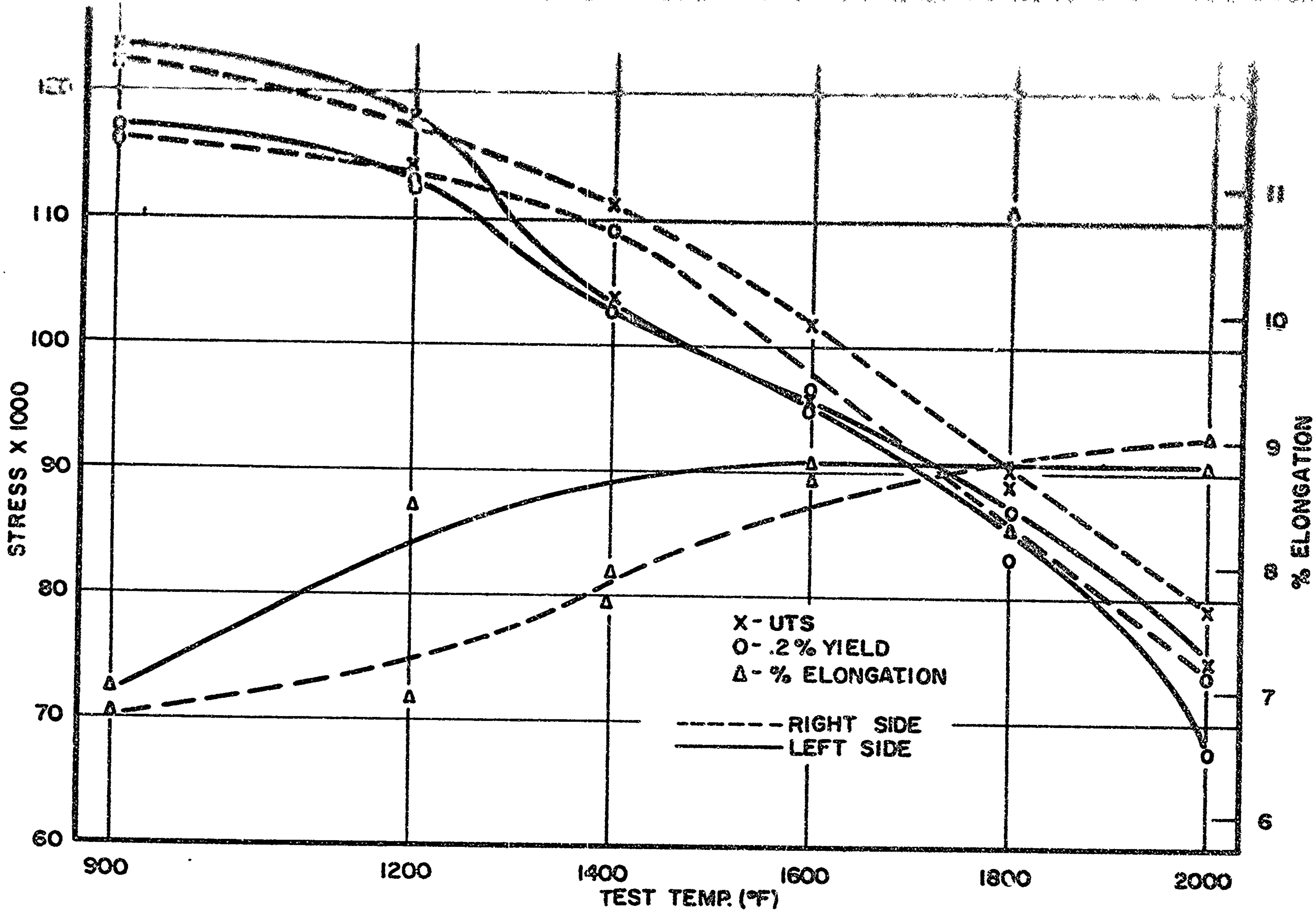


FIGURE 81
TENSILE PROPERTIES FROM 900-2000°F

nominal 120,000 psi to 75,000 psi and the .2% yield showing a corresponding decrease approximately 5,000 psi below the UTS. The elongation, although somewhat erratic, shows a slight increase from a nominal 7% to 9%.

More extensive tensile testing was accomplished on each sheet at 2000°F. Duplicate samples in both the longitudinal and transverse direction were tested from both sides of the sheet. The results of this effort are listed in Table XXXVII.

In reviewing this table, the following results are given:

Maximum Range on Duplicate Samples

	UTS	.2% Yield	% Elongation
	73,800-84,100	68,000-79,600	8.3-9.7
Mean	78,900 ±5,100	73,800 ±5,800	9.0 ±.7

Maximum Range on Samples From One Sheet One Direction

	UTS	.2% Yield	% Elongation
	73,800-84,100	68,000-79,600	7.2-10.3
Mean	78,900 ±5,100	73,800 ±5,800	8.7 ±1.5

Maximum Range on All Samples From One Sheet (Longitudinal and Transverse)

	UTS	.2% Yield	% Elongation
	70,700-84,100	65,600-79,600	6.3-10.3
Mean	77,400 ±6,700	72,600 ±7,000	8.3 ±2

Maximum Range on All Samples From All Sheets

	UTS	.2% Yield	% Elongation
	69,900-84,100	62,900-79,600	6.2-10.8
Mean	77,000 ±7,100	71,200 ±8,300	8.5 ±2.2

TABLE XXXVII

2000°F TENSILE PROPERTIES

Sheet Number	Sample Position	Test Direction	UTS $\times 10^3$	.2% Yield $\times 10^3$	% Elongation
1148-2	Right	Transverse	79.0	74.1	6.5
	Right	Transverse	78.2	72.4	6.5
	Left	Transverse	84.1	79.6	6.3
	Left	Transverse	73.8	68.0	7.6
	Right	Longitudinal	75.1	69.3	7.7
	Right	Longitudinal	74.2	67.9	7.2
	Left	Longitudinal	70.7	65.6	8.0
	Left	Longitudinal	72.7	65.6	10.3
1167-1	Right	Transverse	76.0	69.5	9.5
	Right	Transverse	74.7	68.3	9.6
	Left	Transverse	72.8	69.6	8.3
	Left	Transverse	76.0	69.5	9.7
	Right	Longitudinal	70.7	62.9	9.2
	Right	Longitudinal	72.3	63.5	10.5
	Left	Longitudinal	70.4	66.0	9.7
	Left	Longitudinal	74.0	69.5	10.0
1167-2	Right	Transverse	80.6	76.5	8.3
	Right	Transverse	77.5	73.0	9.9
	Left	Transverse	74.1	63.3	8.3
	Left	Transverse	76.6	71.5	9.3
	Right	Longitudinal	75.9	73.8	10.5
	Right	Longitudinal	70.1	64.7	10.8
	Left	Longitudinal	71.7	66.6	10.5
	Left	Longitudinal	69.9	65.7	9.3

In the transverse direction only, at 2000°F, the allowable variation for specimens in any one lot is as follows:

UTS - ±10% About the Mean

.2% Yield - ±15% About the Mean

In addition, elongation shall not be less than 5%.

From this data, it is shown that comparing both the longitudinal and transverse from two different lots and two gauges, the maximum variations are:

UTS - ±9.33%

.2% Yield - ±11.65%

The minimum elongation is 6.3%.

Considering the transverse direction in one lot, the maximum variations are:

UTS - ±6.47%

.2% Yield - ±7.85%

For the longitudinal direction in one lot, the maximum variations are:

UTS - ±4.12%

.2% Yield - ±4.98%

The values are shown to be well within the desired range.

The tensile properties in the temperature range of 2000° to 3000°F are plotted in Figures 82 and 83. As shown in Figure 82, the strength decreases rapidly in the temperature range of 2200° to 2400°F and then reverts to a more gradual decrease in continuing to 3000°F. The rapid drop in the 2200° to

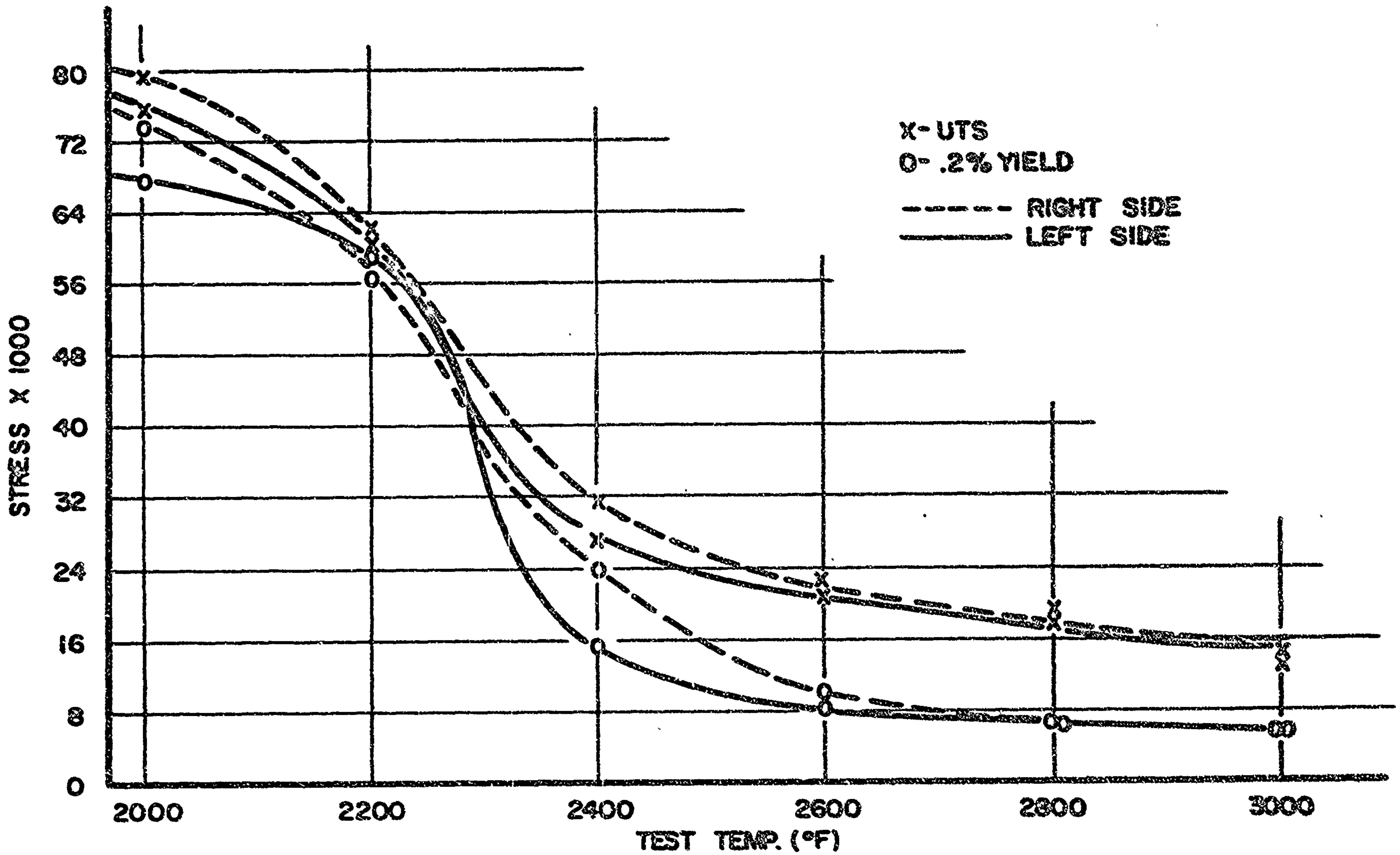


FIGURE 82

TENSILE STRENGTH FROM 2000-3000°F

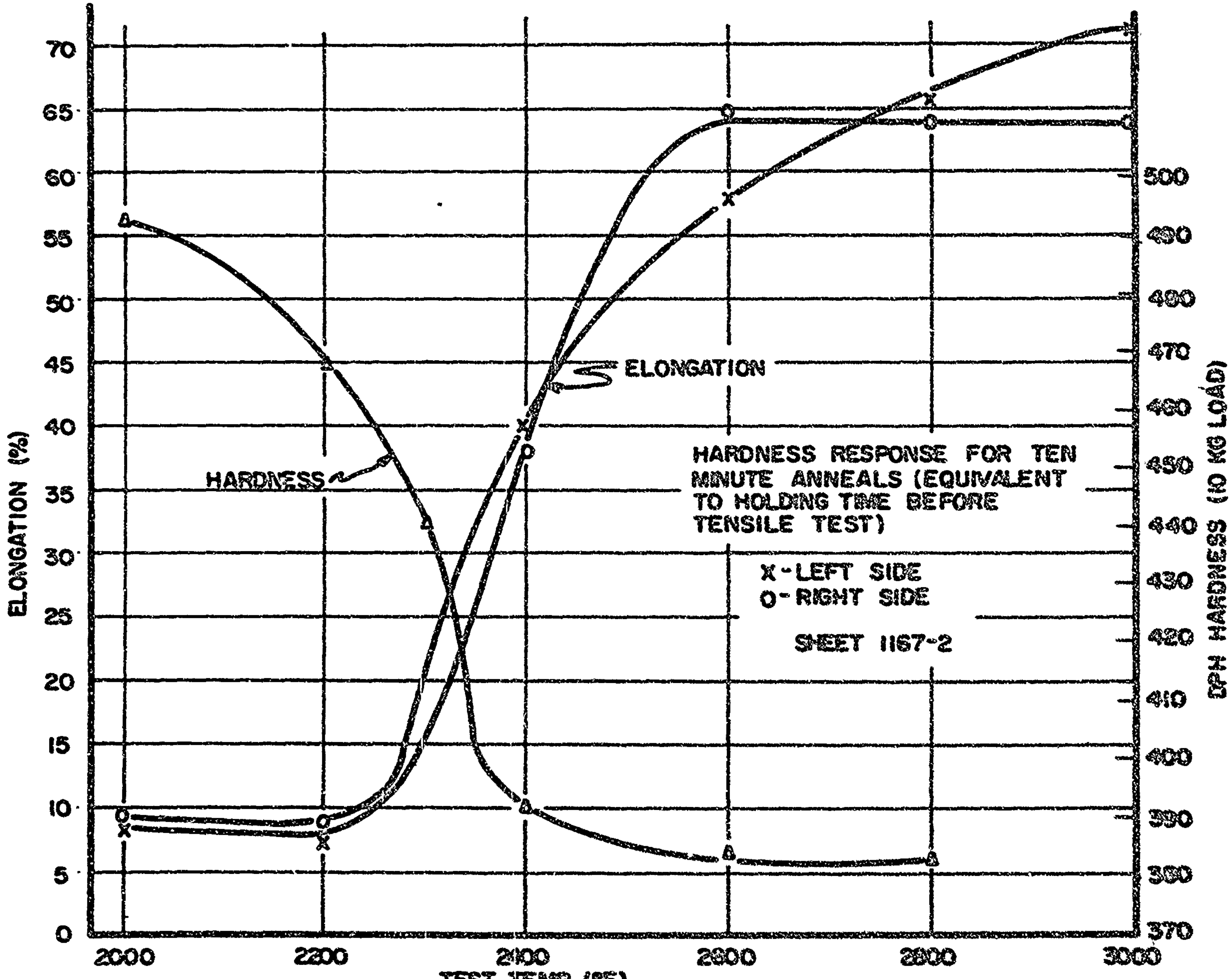


FIGURE 83

TENSILE ELONGATION FROM 2000-3000°F

2400°F range is readily explained by observing Figure 83. It is shown here that in holding the specimen at temperature ten minutes before testing, a corresponding rapid decrease in hardness occurs due to recrystallization. The elongation remaining relatively constant up to the point of initiation of recrystallization shows a rapid increase with increasing recrystallization.

At the 3000°F test temperature, more extensive testing was accomplished. The data shown in Table XXXVIII is for only one sheet (1167-1).

The maximum variation between one set of duplicate samples is:

	UTS	.2% Yield	% Elongation
	12,200-14,000	5,200-6,100	55.7-71.7
Mean	13,000 ±900	5,650 ±450	63.7 ±8

The maximum variation in the transverse direction is:

	UTS	.2% Yield	% Elongation
	12,400-14,800	5,200-6,100	61.6-84.9
Mean	13,600 ±1,200	5,650 ±450	73.2 ±12

The maximum variation in the longitudinal direction is:

	UTS	.2% Yield	% Elongation
	12,200-14,100	5,800-7,400	55.7-77.7
Mean	13,150 ±950	6,600 ±800	66.7 ±11

Using the data listed in Tables XXXV and XXXVII, confidence limits on 900°F and 2000°F tensile properties were calculated using the following:

$$\text{Standard Deviation} - \sigma = \sqrt{\frac{\sum (\bar{x} - x)^2}{n}}$$

TABLE XXXVIII

3000°F TENSILE PROPERTIES
Sheet 1167-1

Sample Position	Test Direction	UTS $\times 10^3$	.2% Yield $\times 10^3$	% Elongation
Right	Transverse	12.4	6.1	84.9
Right	Transverse	12.4	5.2	71.3
Left	Transverse	13.7	5.9	61.6
Left	Transverse	14.8	5.8	62.0
Right	Longitudinal	14.1	5.8	65.1
Right	Longitudinal	12.2	6.1	77.7
Left	Longitudinal	15.3	7.1	71.7
Left	Longitudinal	15.7	7.4	55.7

σ = 68% Confidence Limit

2σ = 95% Confidence Limit

Table XXXIX lists the limits established.

b. .040" Gauge Sheet Product

After determining the rolling characteristics of .060" gauge at the wide width, one sheet bar from heat KD1168 was applied to produce .040" sheet. The as-conditioned size of this sheet bar was 1-3/4" x 4" x 24" long (from direct extruded sheet bar). Following the rolling schedule previously given in Table XXXI, the sheet bar was rolled to .143" thick with no problems. At this point, the resultant sheet product was cut into two pieces with the following dimensions: Piece 1168-1, .143" x 27" wide x 12-3/4" long; Piece 1168-2, .143" x 38-1/2" wide x 12-3/4" long. Cross rolling to .040" gauge was initiated and piece 1168-2 cracked on initial rolling to the extent that a full size .040" sheet could not be produced. It was therefore stopped at .060" gauge to be rolled to .020" with the .020" material. The remaining piece 1168-1 was spread from 27" wide to 36" wide and then cross rolled to .040". As it was slightly undersized to begin with, a full size was not produced. Also, cracking occurred on the trailing end permitting final sheared size of only 34" wide x 24" long.

(1) Bend Transition

The transition temperature for the .040" gauge sheet was determined for three different final stress relief temperatures correlating the left side of the sheet with the right side in both the longitudinal and transverse directions. The data are shown in Table XL.

TABLE XXXIX

CONFIDENCE LIMITS OF 900° AND 2000°F TENSILE PROPERTIES

	Ultimate Tensile Strength x 10^3		.2% Yield Strength x 10^3		% Elongation	
900°F	σ	2σ	σ	2σ	σ	2σ
Longitudinal	114.0 ± 3.9	114.0 ± 7.8	102 ± 6.2	102 ± 12.4	7.1 ± 1.05	7.1 ± 2.1
Transverse	117.8 ± 4.4	117.8 ± 8.8	107 ± 3.8	107 ± 7.6	7.1 ± 1.3	7.1 ± 2.6
Combined L&T	115.9 ± 4.4	115.9 ± 8.8	105 ± 4.6	105 ± 9.2	7.1 ± 1.2	7.1 ± 2.4
2000°F						
Longitudinal	72.3 ± 2.1	72.3 ± 4.1	66.8 ± 3.1	66.8 ± 6.2	9.5 ± 1.2	9.5 ± 2.4
Transverse	77.8 ± 3.4	77.8 ± 6.8	71.3 ± 4.1	71.3 ± 8.2	8.3 ± 1.3	8.3 ± 2.5
Combined L&T	74.7 ± 3.4	74.7 ± 6.8	69.0 ± 4.5	69.0 ± 9	8.9 ± 1.4	8.9 ± 2.7

σ = 68% Confidence

2σ = 95% Confidence

TABLE XL

4T - .040" BEND TRANSITION TEMPERATURE (°F)

Position	Stress Relief Temperature 1600°F	1700°F	1800°F
Longitudinal Left	300	375	350
Longitudinal Right	325	350	325
Transverse Left	250	175	175
Transverse Right	225	250	225

In comparing the left side of each sheet with the right side, four of six are within 25° of each other. Of the remaining two, one shows 50°F difference and the other 75°F difference. Comparison of the three annealing temperatures shows, on the average, decreasing transition with increasing annealing temperature. This correlates to some extent with the data produced on the initial rolling evaluation which indicated that 1700°F was the most desirable annealing temperature and higher bend transitions resulted with anneals above or below this value. In the initial evaluation, the lowest bend transition on .040" gauge was 200°F. Through refinements in the rolling practice, the low values on this material were established at 175°F

(2) Tensile Properties

Using the same parameters previously established for tensile testing, investigations were run to determine the tensile transition temperature and 900°, 2000°, and 3000°F tensile properties. Premature specimen failure prevented accurate determination of the tensile transition temperature. The lowest temperature tests which were satisfactory are summarised in Table XLI. Lower temperature tests than those indicated consistently resulted in specimen failure at one of the supporting pin holes. The 900°F tensile properties are also listed in Table XLI.

TABLE XLI

TENSILE TRANSITION DATA AND 900°F TENSILE DATA (.040" GAUGE)

Position	Stress Relief Temperature (°F)	Test Temperature (°F)	UTS	.2% Yield	% Elongation
Transverse Left	1600	250	190.8	168.6	6.4
Transverse Right	1600	325	173.8	156.2	7.1
Transverse Left	1700	325	173.3	157.5	7.2
Transverse Right	1700	275	182.4	157.7	6.5
Longitudinal Left	1700	300	163.1	142.2	4.7
Longitudinal Right	1700	250	180.7	153.5	6.7
Transverse Left	1800	250	186.3	168.5	7.3
Transverse Right	1800	250	187.6	167.3	6.3
Transverse Left	1700	900	125.2	113.0	5.5
Transverse Left	1700	900	130.0	112.8	4.4
Transverse Right	1700	900	132.6	116.3	4.9
Transverse Right	1700	900	130.3	118.4	4.7
Longitudinal Left	1700	900	116.3	109.2	4.8
Longitudinal Left	1700	900	124.7	115.6	5.2
Longitudinal Right	1700	900	121.0	105.5	5.6
Longitudinal Right	1700	900	120.4	109.8	6.3

The positions indicated as left and right refer to the extreme sides of the sheet. The two different positions were used to determine uniformity within the sheet. Duplicate samples were run for each position with longitudinal left positions showing the greatest variation. Using the mean for these two tests, the variation in UTS is 3.5%. This mean value, 120,500 psi, correlates extremely well with the opposite sheet side which showed a mean value of 120,700 psi. The transverse values also show good correlation between the two sides. Comparing the mean transverse and longitudinal test values, it is shown that the transverse UTS is 8,500 psi greater. The transverse .2% yield is 5,100 psi greater; however, only a 6% elongation exists in the longitudinal direction. This anisotrophy in elongation is attributed to cross rolling even though the strength values which are more sensitive show a slight degree of directionality.

Tensile tests to determine the 2000°F tensile properties were conducted using the same position plan as that for the 900°F tests. Also, as with the 900°F tests, duplicate samples were tested for each position. The results of these tests are listed in Table XLII.

TABLE XLII

2000°F and 3000°F TENSILE PROPERTIES (.040" SHEET)

All Samples Stress Relieved One Hour at 1700°F
All Samples Tested Under a Vacuum of 3×10^{-5} Millimeters

	2000°F		
Position	UTS x 10^3	.2% YS x 10^3	% Elongation
Transverse Left	86.4	81.7	7.2
Transverse Left	86.2	79.7	5.7
Transverse Right	93.1	84.5	5.6
Transverse Right	96.3	84.1	7.1
Longitudinal Left	74.8	71.2	7.5
Longitudinal Left	74.7	66.8	7.2
Longitudinal Right	76.7	71.3	6.9
Longitudinal Right	74.8	67.8	6.4

TABLE XLII (cont.)

	3000°F		
Position	UTS x 10^3	.2% YS x 10^3	% Elongation
Transverse Left	13.4	4.7	54.5
Transverse Left	12.7	4.3	47.7
Transverse Right	12.6	5.0	48.4
Transverse Right	15.0	6.1	52.9

The duplicate samples show extremely good uniformity. The greatest variation between the two sides of the sheet exists in the transverse UTS, 8,400 psi deviation between the two mean. The longitudinal values show a much closer correlation between sides with a deviation of only 1,000 psi UTS. The elongation values are consistent, with a mean variation of 0.6% between longitudinal and transverse. Note that this is identical to the difference in the 900°F elongation values.

3000°F tensile properties were determined in the transverse direction only since the samples recrystallized during heating to test temperature, and the sheet directionality is thus destroyed. The data established is also presented in Table XLII.

c. .020" Gauge Sheet Product

From the rolling characteristics of .060" gauge at the wide widths, two additional sheet bars were applied to produce .020" sheet from Heat KD1147. The nominal starting sizes of these pieces were as follows:

1147-1	2" x 3-1/2" x 20"	Press Forged
1147-2	2" x 3-1/2" x 20"	Press Forged

In addition, one piece of 1148-1, which broke at an intermediate gauge in rolling to .060", was rolled in this series to .020". The rolling schedules established for this material were previously given in Table XXXII.

All but one piece rolled satisfactorily to the intermediate gauge of nominal .110" to .140" thick. The one piece (1147-1) cracked severely on the first pass after the recrystallization anneal at .300" thick. It had been planned to roll two pieces of .020" from this sheet; however, after the cracks were sheared out, it was possible to roll only one piece. A review of the sheet sizes at this point was as follows:

1147-1	.104" x 36-1/4" wide x 14-1/2" long
1147-2	.125" x 38" wide x 20-3/4" long
1148-1	.100" x 39" wide x 10-1/2" long

For further rolling all pieces were packed between AISI 1095 steel. The furnace temperature utilized was 1400°F. All of the material rolled to .060" satisfactorily and, after conditioning, the following pieces were available for rolling to .020":

1147-1	.060" x 37-1/2" wide x 16" long
1147-2	.060" x 48-1/5" wide x 14-3/4" long
1147-2	.060" x 38-1/4" wide x 14-3/4" long
1148-1	.060" x 37-1/2" wide x 16" long
1168-2	.060" x 38" wide x 12" long

For final rolling to .020" it was planned to roll 1147-1 and 1148-1 in one pack and the two pieces of 1147-2 in a second pack with 1168-2 rolling single. In transporting the material to the mill, 1147-1 cracked 2-1/2" in on the edge which required shearing to 35" wide. 1147-1 and 1148-1 were therefore rolled separately as they did not match in size. Again, all pieces were cover plated with AISI 1095 steel. All pieces rolled satisfactorily to .020" gauge.

The last processing operation on the .020" sheets was shearing to size. During this operation, one piece (1147-1) cracked and required reshearing to a shorter length. The final sizes of the five sheets after shearing were as follows:

1147-1	.020" x 34" wide x 34-1/2" long
1147-2A	.020" x 36-1/2" wide x 38" long
1147-2B	.020" x 36-1/2" wide x 38" long
1148-1	.020" x 36" wide x 42" long
1168-2	.020" x 37" wide x 34" long

The flattening operations were accomplished on a roller leveler in conjunction with a 1200°F gas fired furnace. Only one pass per reheat was attempted since the rolls were not preheated and the exit temperature of the sheets ranged from 300° to 400°F. Three to five passes were required to flatten the sheet to within 4% as determined by MAB standards.

For descaling, the following sequence was used:

1. Preheat to 500°F
2. Hydride (1000°F)
3. Air Cool
4. Water Rinse
5. Sulfuric Acid Bath
6. Water Rinse
7. Permanganate Bath
8. Sulfuric Bath
9. Water Rinse

The five sheets were supported vertically on a steel rack for processing through the above sequence. Figure 84 shows a typical sheet at .020". The stains which are prevalent over the sheet surfaces were readily removed by scrubbing.

(1) Inspection

All the sheets including the two which were cracked, were measured for gauge control. Using the MAB standard of one-half AMS 2242, the allowable variation on .020" sheet is ±.0015". The gauge readings from these sheets are summarized in Table XLIII. As shown, only one of the five sheets is within the desired tolerance. The remaining four are all out on the high side varying from .0006" to .0015" over the maximum limit. Sheet 1148-1 could readily be brought within tolerance by pickling off

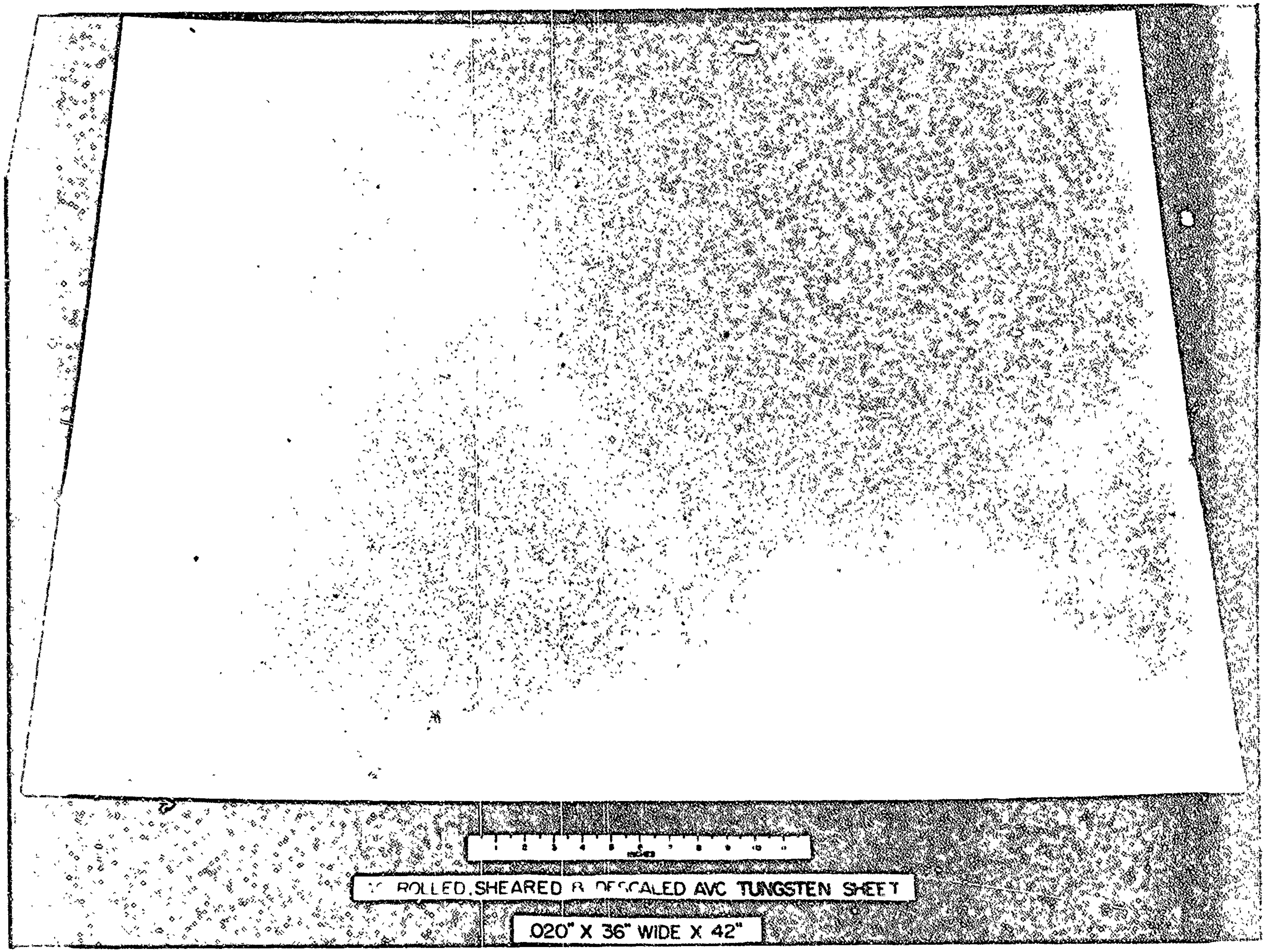


Figure 84

Typical .020" Sheet

TABLE XLIII

GAUGE VARIATION ON .020" SHEET
(Measured 1" From Edge)

	1147-1	1147-2A	1147-2B	1148-1	1168-2
Right Front	.0185	.0190	.0198	.0195	.0185
Right Middle	.0195	.0188	.0211	.0196	.0190
Right Rear	.0194	.0185	.0210	.0195	.0185
Front Middle	.0202	.0220	.0206	.0208	.0195
Rear Middle	.0221	.0225	.0230	.0212	.0198
Left Front	.0207	.0200	.0222	.0214	.0189
Left Middle	.0217	.0202	.0225	.0223	.0193
Left Rear	.0217	.0200	.0223	.0219	.0200
Maximum	.0221	.0225	.0230	.0223	.0200
Minimum	.0185	.0185	.0198	.0195	.0185
Average	.0205	.0201	.0216	.0208	.0192
Out of Tolerance	-.0000	-.0000	-.0000	-.0000	-.0000
	+.0006	+.0010	+.0015	+.0008	+.0000

.0009" since the low reading on this sheet was .0010" above the lower tolerance limit. Sheet 1147-2B which has the highest out of tolerance value could be brought to within .0002" of the high tolerance limit by pickling off .0013" which would put the low value on the lower tolerance limit. The remaining two sheets could not be changed since they both have minimum values right on the lower tolerance limit.

(2) Bend Transition

One sheet bar originally scheduled for .060", which cracked on initial rolling, was reapplied to .020". Another piece originally scheduled for .040" which cracked during the second rolling operation was also rolled to .020". Since the gauge at which intermediate recrystallization anneal differs for each final sheet gauge, the final .020" sheets were rolled by different practices. The five sheets rolled to .020" and the rolling practice used for each are outlined in Table XLIV.

TABLE XLIV

ROLLING SCHEDULES FOR .020" SHEET

Sheet Number	Recrystallization	Stress Relieve #1	Stress Relieve #2
1147-1	.280"	.120"	.060"
1147-2A*	.280"	.120"	--
1147-2B*	.280"	.120"	--
1148-1**	1.00" + .275"	.120"	.060"
1168-2***	.600"	.300"	.060"

*Original .020" Practice
**Originally Scheduled for .060"
***Originally Scheduled for .040"

The bend transition data for these sheets are listed in Table XLV.

TABLE XLV

4T - .020" BEND TRANSITION TEMPERATURE (°F)

Position	Sheet Number 1147-1	1147-2A	1147-2B	1148-1	1168-2
Longitudinal Left	275	125	250	250	150
Longitudinal Right	250	200	250	300	250
Transverse Left	350	250	300	350	125
Transverse Right	325	225	250	325	175

The first sheet (1147-1) as shown in Table XLIV was rolled to the original .020" schedule except for an additional stress relief at .060" gauge. In comparing this sheet with the two rolled by the original process (1147-2A and 1147-2B), it is shown that the .060" stress relief was detrimental to the bend transition properties. The fourth sheet (1148-1) was rolled by a practice similar to the first sheet and the bend transition properties are shown to be similarly poor. Sheet 1168-2 which was originally scheduled for .040" shows by far the best bend transition. The total reduction from the recrystallization anneal on this sheet was 97%. In the scale-up to 24" x 24" sheet, it was shown that this reduction was excessive, resulting in laminated sheet, however, the additional stress relief at .060" was not previously investigated and this apparently permits higher total reduction without laminating. It is shown that the additional stress relief is advantageous only if the total amount of work from the recrystallization anneal is increased since it was shown to be detrimental when applied to the original process, limited to 93% total reduction. The lowest bend transition recorded was 125°F as compared to 225°F which was the lowest value previously established for .020" sheet.

(3) Tensile Properties

The parameters for tensile testing were as follows:

Specimen 3/4" gauge length and 3/16" width

Strain Rates

900°F and Below

.005"/in/min to .6% Yield

.05"/in/min to Fracture

Above 900°F

.05"/in/min

Testing procedures and test temperature ranges investigated were the same for .020" sheet as that used for .040". In attempts to determine the tensile transition temperature, the same problem of premature failure of the test specimen that was experienced on .040" testing occurred on the .020" tests. The lowest temperature tests which were satisfactory are summarized in Table XLVI. No attempt will be made to analyze this data due to the testing problem.

Table XLVI summarized the 900°F tensile properties. Using MAB184-M as a guide for desired maximum limits on variation in test results, the following criteria were used in evaluating the five .020" sheets. Room temperature variation of all test specimens (900°F tests for tungsten).

UTS - ±7% About the Mean

.2% YS - ±10% About the Mean

The actual variations on these sheets are as follows:

	Mean	Variation
UTS x 10^3	110.5	±7.35%
.2% YS x 10^3	100.0	±9.50%

With the exception of two values on Sheet 1148-1, the variation would have been greatly improved.

TABLE XLVI

TENSILE TRANSITION DATA AND 900°F TENSILE DATA (.020" SHEET)

Sheet	Position	Test Temperature	UTS x 10^3	.2% YS x 10^3	Elongation
1147-1	LL	350°F	163.2	145.6	7.6%
1147-2B	LL	300°F	151.2	141.5	10.7%
1168-2	LR	350°F	135.9	119.3	4.0%
1168-2	LR	325°F	140.7	120.4	3.1%
1168-2	TR	350°F	164.8	150.9	2.3%
1168-2	TL	300°F	179.2	158.7	5.5%
1147-2A	TL	300°F	161.5	148.7	6.5%
1147-1	TL	900°F	111.0	96.2	4.2%
	TL	900°F	111.1	98.0	3.1%
	TR	900°F	113.0	96.3	5.3%
	TR	900°F	110.9	96.1	4.1%
	LL	900°F	102.3	96.3	5.9%
	LL	900°F	105.2	93.4	3.9%
	LR	900°F	104.7	98.9	5.1%
	LR	900°F	103.3	96.8	4.8%
1147-2A	TL	900°F	104.8	93.7	3.3%
	TL	900°F	106.4	97.4	2.4%
	TR	900°F	107.6	98.3	3.2%
	TR	900°F	105.4	91.8	2.9%
	LL	900°F	104.7	91.9	3.6%
	LL	900°F	105.9	92.4	3.6%
	LR	900°F	103.9	90.5	3.1%
	LR	900°F	-	-	-
1147-2B	TL	900°F	110.5	104.5	8.4%
	TL	900°F	108.9	105.7	3.1%
	TR	900°F	110.4	105.7	3.9%
	TR	900°F	-	-	-
	LL	900°F	103.7	96.0	6.0%
1148-1	TL	900°F	107.9	94.3	5.4%
	TL	900°F	111.5	95.1	3.6%
	TR	900°F	117.3	106.8	4.6%
	TR	900°F	118.6	109.5	5.2%
1168-2	TL	900°F	106.9	97.4	3.8%
	TR	900°F	104.3	96.8	4.2%

Tensile properties at 2000°F were determined only in the transverse direction. The results are contained in Table XLVII. The MAB recommended maximum variations are as follows:

UTS - ±10% About the Mean

.2% YS - ±15% About the Mean

The actual variations on the 2000°F test data were as follows:

	Mean	Variation
UTS x 10^3	78.7	5.21
.2% YS x 10^3	68.3	11.42

As shown, the actual variations are much lower than the above desired limits. The specimens for sheet 1148-1 were all broken during sample preparation.

Investigations on 3000°F tensile properties were limited to the transverse direction, but due to recrystallization, directionality is essentially eliminated. The results of the 3000°F tests are contained in Table XLVII. As shown, a considerable strength decrease has occurred from the previous 2000°F tests and this is the result of the aforementioned recrystallization which occurs at the test temperature. Again, test data was not acquired on 1148-1 due to breakage during preparation.

E. Scale-Up to 36" x 96" Sheet From 8" Diameter Extrusion Billet

1. Sheet Bar Application

A total of nine 8" diameter conditioned ingots were direct extruded to sheet bar for application to the final pilot production of 36" x 96" sheet product. The difficulties experienced from the scale-up from 6" to 8" diameter extrusion billet resulting in severe surface defects in the as-extruded sheet bar caused con-

TABLE XLVII

2000°F AND 3000°F TENSILE PROPERTIES (.020" SHEET)

2000°F

Sheet	Position	UTS x 10^3	.2% YS x 10^3	% Elongation
1147-1	Transverse Right	78.9	67.3	5.2
	Transverse Right	74.6	63.9	3.5
	Transverse Left	75.3	60.5	3.6
	Transverse Left	80.2	64.7	4.3
1147-2A	Transverse Right	81.5	74.8	3.1
	Transverse Right	81.9	71.6	3.2
	Transverse Left	80.9	73.0	4.0
	Transverse Left	82.8	76.2	3.3
1147-2B	Transverse Right	79.6	72.1	4.4
	Transverse Right	80.6	73.9	3.7
	Transverse Left	79.2	68.7	3.3
	Transverse Left	78.8	69.0	3.1
1169-2	Transverse Right	78.7	62.7	3.6
	Transverse Right	79.2	65.3	3.5
	Transverse Left	80.2	70.5	3.3
	Transverse Left	80.2	69.1	3.5

3000°F

Sheet	Position	UTS x 10^3	.2% YS x 10^3	% Elongation
1147-1	Transverse Right	14.5	7.0	66.7
	Transverse Right	15.5	3.7	53.5
	Transverse Left	12.7	5.4	56.9
	Transverse Left	13.9	2.5	42.8
1147-2A	Transverse Right	15.3	7.4	48.5
	Transverse Right	13.6	6.2	40.2
	Transverse Left	13.1	5.7	34.5
	Transverse Left	12.8	5.6	49.7
1147-2B	Transverse Right	15.0	6.8	42.3
	Transverse Right	15.0	6.9	43.1
	Transverse Left	16.3	6.9	56.5
	Transverse Left	15.1	6.2	40.0
1168-2	Transverse Right	13.5	7.3	26.3
	Transverse Right	16.4	6.2	26.8
	Transverse Left	14.3	5.0	30.9
	Transverse Left	11.0	5.3	27.1

siderable difficulty in applying the sheet bar sections to a final sheet rolling schedule. In order to acquire as much material as possible for application to the final sheet sizes, severe conditioning by hand grinding was necessary on all of the as-extruded sheet bars. The as-conditioned extrusions were irregular in shape after grinding which limited the effectiveness of ultrasonic inspection. Also, intelligent application of the irregular shapes was made extremely difficult. However, the as-conditioned sheet bars were sectioned into lengths to give the best possibility for a finish sheet product.

2. Process Schedule for 36" x 96" Sheet

The processing schedules for the three sheet gauges required in the pilot production phase of the contract are shown in Table XLVIII. Two principle factors were used in establishing the schedules:

1. The amount of reduction from recrystallization and intermediate stress relief down to final gauge.

2. The ratio of cross rolling after the recrystallization anneal.

The desired percent reduction after the above two anneals was 93% to 82% respectively and the cross rolling ratio was 1:1. These conditions could not be met exactly because of the thick sheet bar cross section and the actual conditions are shown in Table XLVIII.

3. Pilot Production Rolling of 36" x 96" Sheet

The larger size sheet bar produced for the pilot production requirements of the contract required considerable modification to the pi[illegible] rolling operation. Initially to produce a .060" x 36" x 96" sheet weighing 145 pounds, a starting sheet

TABLE XLVIII

ROLLING AND ANNEALING SCHEDULE
REDUCTION SCHEDULE AND CROSS ROLLING RATIO

Sheet Bar 2" x 6" x Width*

Process	Temperature (°F)	Gauge .060	Gauge .040	Gauge .020
First Rolling	2300	.800	.540	.280
Recrystallize	2800			
Second Rolling	2300	.360	.200	.110
Stress Relieve	2000			
Third Rolling	2000	.120	.120	--
Fourth Rolling	1400	.060	.040	.020
Reduction From Recrystallization		92.5%	92.7%	93.0%
Reduction From Stress Relief		83.5%	80.1%	81.7%
Cross Rolling Ratio		1.52:1	1.27:1	1.34:1

*Widths For the Various Gauges Are as Follows:

.060" - 32"
.040" - 23-1/2"
.020" - 25-3/4" (Two Pieces From Each Sheet Bar)

bar size was required with a nominal weight of 270 pounds. This required a sheet bar with a physical dimension of approximately 2" x 6" x 32". The sheet bar extrusion from Heat KD1231 was sectioned into two pieces and piece 1 was applied for initial rolling investigation. After heating to 2300°F, it was rolled one pass 31" wide. However, with this width the reduction was approximately half of the desired .2". After reheating the second pass was made again attempting to take .2" reduction but only .125" was achieved. Due to the limited reduction on the second pass, the entire sheet bar alligatored (cracked in the longitudinal plane over the entire length and width). The cracking problem could also be partially attributed to the low reduction ratio on the extrusion (3.35:1) which resulted in an extremely coarse grain structure and limited degree of work in the center.

The second section of sheet bar KD1231 had a longitudinal crack approximately 2" from one edge, which required cutting the bar width from 6" down to 4". This second sheet bar then had dimensions of 2-1/8" x 4" x 24-3/8" which was borderline for producing a full size .040" thick sheet. As previously mentioned, the problems associated with the first sheet bar appeared to relate to the limiting mill capacity and the extrusion practice which resulted in coarser grain structure than desired. The maximum reduction per pass that can be achieved on a given mill facility is directly proportional to the rolling width of the sheet bar, which, in the case of the first bar, was 31". Although the second sheet bar was narrower in width, at 24-3/8", and considered within the existing mill capacity (based upon the successful rolling of 2" thick by 20" wide sheet bar in previous investigations), it was decided to modify the rolling practice in order to gain information to apply to future rolling of the required 31" widths.

The second bar was therefore rolled in the same direction as it had been extruded. For example, in the 4" rolling width, which permitted accomplishing the desired reductions per pass. In two passes, the sheet bar was reduced from 2-1/8" thick to a nominal 1-1/4" thick with no evidence of cracking. At this point, it was recrystallized by annealing one hour at 2800°F. For the second rolling operation, the piece was rolled 38" wide to a nominal .6" thick. On the first pass, a very slight alligator type crack occurred on the leading edge and the piece was reversed for subsequent passes, which put the cracked leading edge on the trailing edge. No additional cracking was observed during the rolling operation; however, ultrasonic examination at .6" thick showed that limited cracking was present, extending in from both the leading and trailing edges. The available mill capacity was again the limiting factor, in that this type of cracking is generally associated with low reductions per pass.

The remaining eight sheet bars in which the extrusion ratio had been increased to 4.25:1 were applied for rolling according to the schedule given in Table XLVIII. The major problem areas which developed during the final rolling operations can be summarized as follows:

1. During initial breakdown of the nominal 2" thick sheet bar, alligator type cracking still occurred. This problem was minimized but not completely eliminated by increasing the reduction per pass. For example, it was necessary to crop off an average of 1" from the leading and trailing ends of .800" gauge as-rolled sheet for a resultant 13% loss.

2. At .120" gauge, just prior to final rolling, the sheets were as large as 30" x 30" and

required cutting all edges in addition to complete surface conditioning. These operations frequently resulted in severe cracking problems.

3. After the final rolling operation, the sheets normally had edge cracks up to 1" resulting from the use of the AISI 1095 cover sheets. In addition, the sheets were usually out of flat in the as-rolled condition. Subsequent hot shearing operations to remove the edge cracks and as-rolled ends frequently resulted in edge crack propagation due to the combination of the existing cracks and the stresses incurred in shearing an out of flat sheet.

4. In attempts to correct the out of flat condition off the rolling mill, roller leveling was attempted, but the handling problems and the notch-sensitive effect of the edge tears in some cases initiated crack propagation during this operation.

5. Handling of the finish rolled and sheared sheet through stress relieving and finish inspection operations also resulted in an occasional cracking situation.

Four of the nine original extrusions applied were totally scrapped or removed from the rolling schedule due to various combinations of the problems mentioned above. The remaining five extrusions were applied and rolled to final sheet. The final sheet sizes produced throughout the program are given in Appendix V.

As the above appendix will show, the problems associated with melting, extrusion and rolling prohibited the attainment of the contract objectives of a 36" x 96" sheet in any of the three gauges under investigation.

4. Evaluation of Final Rolled Sheet Product

a. Hardness Uniformity and Response to Heat Treatment

Samples were taken from all final rolled sheet product in order to determine hardness uniformity and response to heat treatment to various annealing temperatures. Photomicrographs were prepared for visual observation and estimation of percent recrystallization at the various annealing temperatures studied. The samples were annealed at 2000°, 2100°, 2200°, and 2300°F for one hour at temperature. Table XLIX gives the hardness values and estimated percent recrystallization at the various annealing temperatures and Figure 85 shows a plot of average hardness for each gauge versus response to heat treatment.

From Table XLIX and Figure 85, it can be seen that complete recrystallization was not obtained at the highest annealing temperature investigated. From the slope of the average hardness curve, it is estimated that the 100% recrystallization would be obtained on the .040" and .060" gauge sheet at approximately 2400°F. For the .020" gauge material, the 100% recrystallization temperature would be between 2300° and 2350°F. The average hardness curves also indicate that the .040" and .060" gauge material are similar in their response to heat treatment while the .020" gauge material exhibits a recrystallization rate of approximately 100°F lower than the other two gauges. This is relatively consistent with the data established in earlier phases of this report. The hardness variation from sheet to sheet, as given in the table, and

TABLE XLIX

HARDNESS AND PERCENT RECRYSTALLIZATION VERSUS HEAT TREATMENT AT VARIOUS GAUGES

		Heat Treatment Temperature							
		2000°F		2100°F		2200°F		2300°F	
Sheet Number	Gauge	Hardness	% Recryx	Hardness	% Recryx	Hardness	% Recryx	Hardness	% Recryx
KD1287-2	.060	491	0	481	5	469	50	397	90
KD1287-2*	.060	475	0	476	10	447	60	395	95
KD1288-1	.060	500	0	484	2	446	40	395	95
KD1289-3	.060	466	0	462	5	433	50	399	90
KD1289-3*	.060	471	0	465	5	416	75	385	95
KD1290-1	.060	481	0	469	2	433	10	404	50
KD1289-4	.040	485	1	463	5	426	35	404	70
KD1291-1	.040	501	1	500	3	448	10	419	50
KD1291-2	.040	512	1	476	15	436	75	389	95
KD1291-3	.040	488	10	484	35	451	75	409	95
KD1291-4	.040	479	10	476	35	392	80	383	98
KD1289-5	.020	461	10	431	30	410	75	367	95
KD1291-6	.020	481	15	430	60	388	90	373	98

*Opposite End

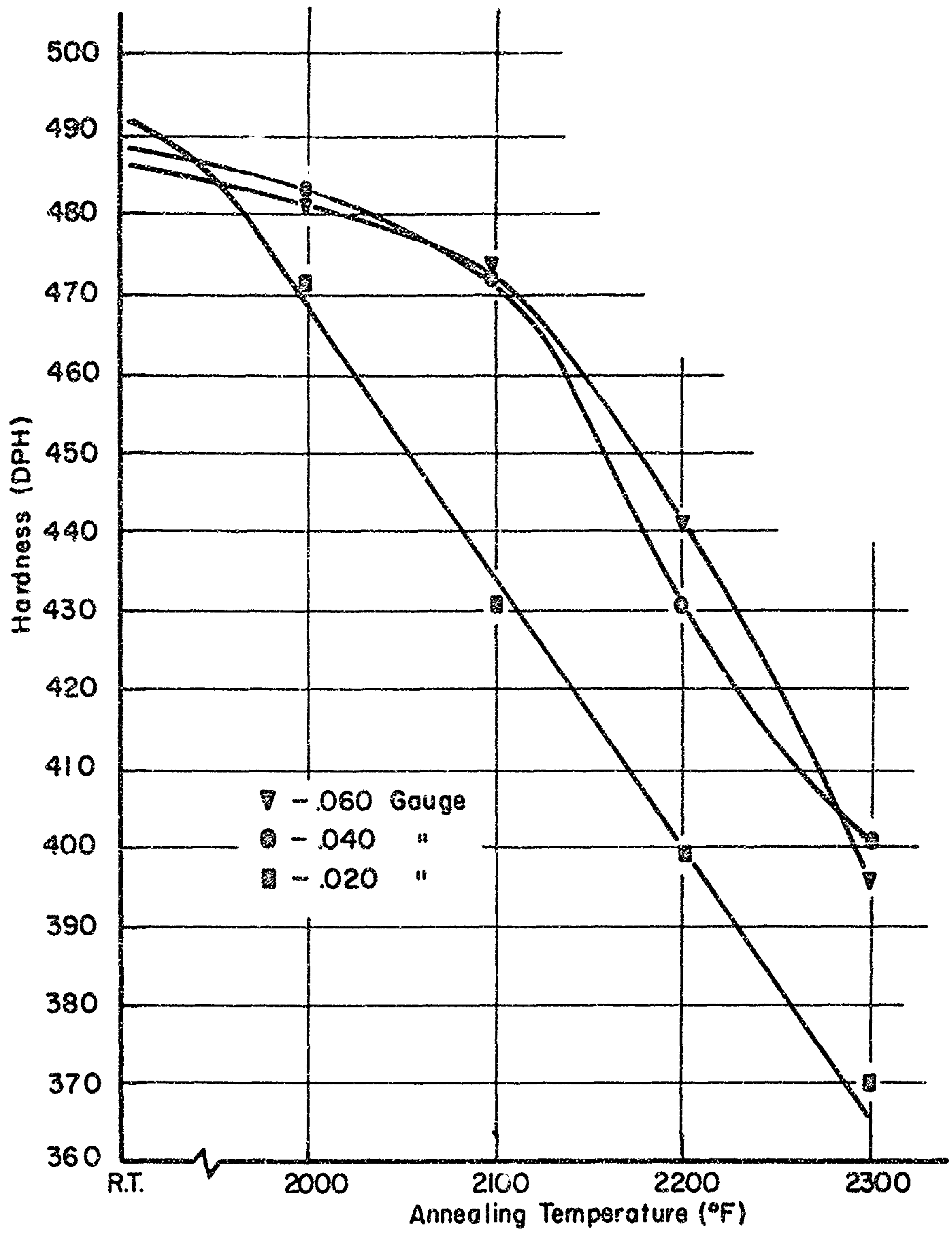


FIGURE 85

AVERAGE HARDNESS VS RESPONSE TO HEAT TREATMENT – FINAL PILOT PRODUCTION SHEET

based on the same gauge, is relatively uniform considering that processing changes were necessary, in some cases, in order to acquire maximum size finished sheet.

b. Bend Transition Temperature

Bend transition temperatures were determined on the product of each sheet bar applied to the three final rolled gauges. In order to demonstrate uniformity within each given sheet, opposite end samples were taken from sheet produced from the original sheet bar even though, in some cases, due to cracking or breakage in handling, more than one sheet was represented. The bend transition data from the pilot sheet product is given in Table L.

The bend transition temperatures were determined as previously described using a 4T bend radius. However, following more recent recommendations by MAB, the ram speed was changed from 8" per second to 1" per second. The lowest bend transition temperature was 150°F recorded on .060" gauge material in the longitudinal or final roll direction. Although the transverse bend transition temperatures varied from 275° to 425°F, at no time was the bend transition higher than 425°F in any gauge tested. The average bend transition for all gauges of sheet when comparing opposite ends shows fairly good uniformity. Average longitudinal bend transition showed a spread of 10°F and the transverse bend transition showed a spread of 20°F in comparing opposite end samples. However, there was considerable scattering of transition data within the same gauge application and utilizing the same processing schedules. This scattering of test data could be due to a number of causes, most probable of which are minor rolling and annealing variations, and sample preparation methods.

TABLE L

BEND TRANSITION TEMPERATURE OF PILOT SHEET PRODUCT

Sheet Number	Gauge	Bend Transition Temperature Longitudinal	Bend Transition Temperature Transverse	Bend Transition Temperature (Opposite End of Sheet) Longitudinal	Bend Transition Temperature (Opposite End of Sheet) Transverse
KD1287-2	.060	175	275	225	425
KD1288-1	.060	200	350	275	375
KD1288-2	.060	150	375	--	--
KD1289-3	.060	200	425	225	425
KD1290-1	.060	250	325	275	325
Average	.060	195	350	250	362
KD1289-4	.040	250	375	225	300
KD1291-1	.040	225	325	275	300
KD1291-2	.040	300	300	225	350
KD1291-3	.040	325	325	225	400
KD1291-4	.040	225	425	200	400
Average	.040	260	350	230	350
KD1289-5	.020	250	325	275	300
KD1291-6	.020	250	325	250	400
Average	.020	250	325	262	350
Average of All Gauges		233	344	243	364
Range of All Gauges		150/325	275/425	200/275	300/425

c. Tensile Properties

Tensile properties were determined at 900°F on the final gauge of each sheet bar applied. The results are given in Table LI. Also given in the table is a short summary of process history on each individual sheet bar since some reapplication and process changes were necessary due to breakage during rolling and handling. Tensile specimens were prepared according to the configuration given in Figure 47 using EDM methods. No particular problems were noted in the machining of the tensile blanks. The edges of all specimens were deburred and finish ground to the specimen tolerances required.

Direct correlation could not be made when comparing results from the various gauges with the processing history of the various sheets. The longitudinal tensile strength varied from 89,700 psi to 119,500 psi. The transverse tensile results varied from 92,200 psi to 128,000 psi. The .2% yield strength, as would be expected, varied directly with the ultimate tensile results. Percent elongation measurements on a few of the sheets were considerably below the average. This variation could not be ascribed to processing history and must be considered due to sample preparation or misalignment in the test rig. Very little variation was noted in the percent elongation values for the longitudinal versus the transverse tensiles. The average percent elongation of both longitudinal and transverse samples, excluding the low values was approximately 8%. These values are consistent with the values found in the data developed in the scale-up portions of the contract.

d. Final Inspection

All finish sheet produced from the pilot production run were final inspected for average gauge variation, finish sheet size, flatness, surface finish, and disposition. The

TABLE LI

900°F TENSILE PROPERTIES OF PILOT SHEET PRODUCT

Sheet Number	Gauge	Ultimate Tensile Strength (x 10^3 psi) Longitudinal	Ultimate Tensile Strength (x 10^3 psi) Transverse	.2% Yield Strength (x 10^3 psi) Longitudinal	.2% Yield Strength (x 10^3 psi) Transverse	% Elongation Longitudinal	% Elongation Transverse	Process History
KD1287-2	.060"	108.5 116.1	117.3 121.1	97.9 104.1	113.1 110.3	8.5 8.8	8.0 8.8	Straight Rolled .800" to .225" Cross Rolled to .060"
KD1288-1	.060"	114.3	119.5	104.3	107.9	7.5	4.4	Straight Rolled .800" to .225" Cross Rolled to .060"
KD1289-3	.060"	118.0 111.8	120.3 117.6	109.4 100.1	106.6 110.0	7.6 13.5	8.3 10.6	Straight Rolled to .360" Cross Rolled to .060"
KD1290-1	.060"	89.7	100.7	78.9	95.7	2.1	2.7	Straight Rolled to .360" Cross Rolled to .060"
KD1289-4	.040"	98.8	99.4	93.1	93.2	7.5	5.0	Straight Rolled .540" to .200" Cross Rolled to .040"
KD1291-1	.040"	102.6	102.1	91.8	94.9	2.4	9.2	Cross Rolled .540" to .200" Cross Rolled .200" to .040"
KD1291-2	.040"	109.4	117.2	102.8	107.9	7.6	7.5	Cross Rolled .540" to .200" Cross Rolled .200" to .040"
KD1291-3	.040"	100.5	92.2	87.1	80.7	12.6	13.0	Cross Rolled .540" to .200" Cross Rolled .200" to .040"
KD1291-4	.040"	103.6	106.7	88.3	94.8	7.3	7.8	Straight Rolled .540" to .300" Cross Rolled to .040"
KD1289-5	.020"	103.1	111.2	93.6	86.8	6.6	3.5	Cross Rolled .280" to .120" Cross Rolled to .020"
KD1291-6	.020"	119.5	128.8	114.1	116.9	2.7	4.8	Cross Rolled .280" to .020"

recommended allowable gauge tolerances as given by MAB were to conform to one-half of AMS 2242. From these recommendations, the allowable tolerance in .060" sheet was ±.003, on .040" sheet ±.0025, and on .020" sheet ±.0015. The MAB recommendation for flatness is maximum 4% out of flat. Table LII gives the gauge variation and flatness determinations on all finish rolled sheet. From Table LII, six out of 21 sheets were either rolled under-gauge or sections of the sheet were under-gauge according to the MAB tolerance limits. In addition, three of the 21 sheets were out of gauge tolerance. For example, the deviation from gauge over the sheet is greater than the plus or minus percent tolerance. A total of 11 sheets out of the 21 were over-gauge but within the tolerance limits which means that they could be brought into gauge by a pickling operation.

The problems associated with proper gauge control were caused by 1) the necessity for rolling in a pack to acquire finish gauge, 2) difficulty in determining the exact gauge due to oxide build-up on the sheet, and 3) the necessity for intermediate conditioning of the sheet surface prior to the final rolling operation.

Flatness determination indicated that five out of the 21 sheets were above the 4% maximum out of flat tolerance recommended by MAB. Examination of the finish sheet sizes in the table indicate that the out of flat condition, in general, varies with the increased width of the sheet. The wider and larger the sheet dimensions, the greater is the measured out of flat condition. Four of the 21 sheets exhibited a poor surface condition due to the excessive conditioning operations necessary prior to rolling.

The overall results of the final inspection indicated that only two sheets met the specifications as outlined, with eleven additional sheets being able to meet specification

TABLE LII

FINAL INSPECTION OF FINISHED SHEET FROM PILOT PRODUCTION RUN

Heat Number	Applied Gauge	Average Gauge	Gauge Variation	Finish Size	% Out-of-Flat (AMS 2242)	Surface Finish	Disposition
KD1287-2	.060"	.0564"	+.0016" -.0014"	.0564" x 7" x 23-1/2"	0	Good	Under-Gauge
		.05[illegible]	+.0028" -.0017"	.0582" x 12" x 48"	1.2	Good	Under-Gauge
		.0586"	+.0019" -.0026"	.0586" x 9-1/4" x 18-3/8"	2.0	Good	Under-Gauge
KD1288-1	.060"	.0523"	+.0012" -.0013"	.0523" x 25-1/2" x 50-3/4"	4.4	Good	Under-Gauge Out of Flat
KD1288-2	.060"	.0585"	+.0015" -.0015"	.0585" x 7-1/2" x 13-3/8"	1.3	Good	OK
KD1289-3	.060"	.0595"	+.0015" -.0020"	.0595" x 17-1/4" x 17-3/4"	6.5	Good	Out of Flat
		.0572"	+.0018" -.0012"	.0572" x 9-11/16" x 23-5/8"	1.5	Good	Under-Gauge
		.0576"	+.0014" -.0026"	.0576" x 10" x 22-1/8"	1.5	Good	Under-Gauge
KD1290-1	.060"	.063"	+.001" -.002"	.063" x 21-1/2" x 38-1/2"	2.2	Good	Over-Gauge OK With Pickling
		.0628"	+.0022" -.0018"	.0628" x 21-3/8" x 16-1/8"	1.8	Good	Over-Gauge OK With Pickling
KD1289-4	.040"	.0437"	+.0013" -.0017"	.0437" x 24" x 72"	4.1	Good	Over-Gauge OK With Pickling
KD1291-1	.040"	.0425"	+.0025" -.0025"	.0425" x 12-1/4" x 23-3/8"	2.2	Good	Over-Gauge OK With Pickling
		.0421"	+.0019" -.0041"	.0421" x 17-1/2" x 24"	1.5	Good	Out of Gauge Tolerance
KD1291-2	.040"	.0415"	+.0015" -.002"	.0415" x 18-1/2" x 21"	.5	Good	Over-Gauge OK With Pickling
		.0417"	+.0023" -.0022"	.0417" x 24" x 30"	2.0	Good	Over-Gauge OK With Pickling
KD1291-3	.040"	.0412"	+.0018" -.0042"	.0412" x 19-1/4" x 21-1/4"	4.1	Good	Out of Gauge Tolerance Out of Flat
		.0401"	+.0024" -.0021"	.0401" x 19-5/8" x 23-1/2"	3.6	Good	OK
KD1291-4	.040"	.043"	+.0020" -.0015"	.043" x 10" x 35"	3.5	Good	Over-Gauge OK With Pickling
		.0431"	+.0019" -.0016"	.0431" x 1[illegible]-1/2" x 23"	1.8	Poor	Over-Gauge
		.0448"	+.0012" -.0013"	.0448" x 16" x 20"	.6	Poor	Over-Gauge
		.044"	+.001" -.001"	.044" x 10-1/2" x 15-1/2"	.5	Good	Over-Gauge OK With Pickling
KD1289-5	.020	.0209"	+.0016" -.0044"	-----	3.3	Good	Out of Gauge Tolerance
KD1291-6	.020"	.0229"	+.0006" -.0014"	.0229" x 14-1/4" x 37-1/4"	2.8	Poor	Over-Gauge
		.0206"	+.0029" -.0036"	.0206" x 16" x 20"	5.3	Poor	Out of Gauge Tolerance Out of Flat

following a gauge removal pickle. Due to the inability to attain the contract objectives on sheet size, a gauge removal pickle operation was not initiated since additional handling would be involved.

V. Summary

A. Ingot Melting

From recommendations made by the state-of-the-art survey, pure unalloyed arc-cast tungsten was chosen as the candidate material for the sheet rolling program. In order to acquire the highest and most consistent purity levels in tungsten ingot, tungsten powder produced from the hydrogen reduction of ammonia paratungstate was used as the raw material source. One powder lot was utilized throughout the entire program to insure reproducibility of results. In order to control oxygen content of the electrode and to insure maximum consolidation by isostatic compaction and hydrogen sintering, a tungsten powder lot with a particle size range to give a 3.35 micron average and a bulk density of 68.6 grams per cubic inch was selected. The raw material, in the form of pressed and sintered electrode bar, was purchased to Universal-Cyclops Specification WEB 61-3-A given in Appendix II of this report.

Due to problems in handling the electrode bar in the green state, the suppliers held the length to diameter ratio of the bar at 16:1 maximum. All electrode bars were supplied to a minimum density requirement of 90% of theoretical. Machine threaded joints were used for connecting the electrode segments utilizing powder metallurgy tungsten bar stock as nipple connectors.

Optimum electrode diameter to mold diameter ratios to acquire the highest yield ingot to conditioned billet was determined to be in the range of .450 to .50. Overall ingot purity was excellent with carbon and oxygen giving the greatest variation in the analysis. Average oxygen analysis was 11 ppm; average carbon analysis was approximately 28 ppm.

In order to scale-up for melting of larger diameter ingots (i.e. 6" diameter, 8" diameter, and 9 1/2" diameter) modifi-

cations were necessary to the arc melting furnace in the form of additional power input and modification of the cooling system to give increased cooling effect. The increased electrode weight necessary to scale-up to the larger diameter ingots resulted in a notch-sensitive effect in the thread section of the electrode causing cracks during the melting operation. This situation was virtually eliminated by incorporating a slight radius on the thread root section. Localized sidewall porosity was evident on all ingot diameters with the worst condition being present on the largest diameter ingot (9-1/2" diameter) which resulted in poor yields and excessive conditioning. The problem was ascribed to erratic melt conditions caused by 1) electrode camber and alignment in the furnace, 2) variations in electrode density and resultant current flow through the electrode, and 3) limitations in total available power for the melting operation. Cracks developed in two of the eleven 9-1/2" diameter ingots melted for the pilot production phase of the program. These cracks propagated throughout the length of the ingot and apparently proceeded upward from the pad. It was determined that the cracks propagated either during ingot solidification or from thermal expansion and contraction during annealing.

B. Ingot Breakdown

Primary working of the conditioned arc-cast ingot was evaluated using both direct forging and extrusion techniques. Direct forging attempts resulted in peripheral cracks on both upset forged and radial forged billet sections. The forging evaluation indicated that successful forging could be accomplished by utilizing a small initial reduction (20%) with an intermediate recrystallization anneal between forging operations.

Since cc -iderably more experience has been developed in industry on extrusion techniques for primary breakdown of tungsten ingot rather than direct forging, it was concluded that extrusion

offered the greatest potential towards meeting the breakdown objective. A total of six nominal 3" diameter conditioned ingots were applied for the initial extrusion evaluation. Three of the billets were extruded to 1.5" diameter and the remaining three were extruded to a .6" thick x 2" wide sheet bar configuration. The reduction ratio used for the rounds were 4.5:1 and for the sheet bar configuration 6.6:1. Higher temperatures were used for the sheet bar extrusion due to the higher reduction ratio. All billets were extruded successfully, with the rounds indicating a maximum yield at 4.5:1 reduction at a 3000°F furnace temperature, and the sheet bar configuration with a 6.6:1 reduction ratio at a 3150°F furnace temperature. Slight surface tears were evident on all the extrusions but no correlation could be made with surface condition versus extrusion temperature. Press forging evaluation of the extruded rounds were made in order to correlate the results with that of the direct extruded sheet bar configuration.

The as-extruded rounds were press forged to a 3/4" x 2" sheet bar from a 2300°F furnace temperature which resulted in a forging temperature in the range of 1925° to 1975°F. The press forging operation resulted in slight edge cracking on all three forgings. The yield from conditioned extrusion to condition forging resulted in an approximate 20% loss of material.

Two additional 1-1/2" diameter round extrusions were prepared for InFab forging studies. These extrusions were threaded on one end to facilitate holding during impact forging. Initial heating temperatures in the range of 4000°F were utilized but rapid heat loss was experienced due to the small mass and the high thermal conductivity. The forgings had an irregular cross section which was attributed to excessive reductions per impact. The two extrusions were successfully reduced to 1/2" thick sheet bars for further rolling studies.

The scale-up of extrusion from 3" diameter to 4" diameter conditioned billet using initial extrusion parameters established resulted in severe longitudinal cracks in the extrusion. It was determined that the cracks were caused by the increased transfer time from furnace to press and the furnace temperature was increased from 3050° to 3500°F before sound extrusions could be produced. At this temperature, one of the 4" diameter conditioned billets were extruded directly to sheet bar with no apparent problems. Slight surface tearing was evident on all extruded bars. Press forging of the round extrusions to sheet bar configuration was again accomplished successfully with a resultant yield loss of approximately 8%.

However, since direct extrusion to sheet bar was accomplished satisfactorily, the increased yield and the elimination of the forging step prompted extrusion of 8" diameter conditioned billet to be direct extruded to sheet bar. In the scale-up for extrusion from 8" diameter conditioned ingot, the extrusion ratio was dropped to 3.35:1 due to the marginal capacity of the available extrusion press. The 8" diameter ingot was extruded to a 2-1/2" x 6" sheet bar configuration. The resultant extrusion exhibited severe surface tears and ultrasonic examination revealed a crack running longitudinally along the extrusion for a distance of approximately 12" x 1/2" in depth. The crack and surface tears were attributed to the lower reduction ratio. The reduction ratio was increased to 4.25:1 and furnace temperature was increased to compensate for the increased extrusion pressure. Severe surface tears still persisted in the extruded sheet bars and subsequent extrusions made with decreasing furnace temperature relieved the surface problem somewhat but did not eliminate it. The severe surface tears present in the sheet bars for the final pilot production run resulted in very heavy conditioning losses, with the resultant problem that application to the rolling operation was very difficult.

C. Sheet Rolling

Three direct extruded sheet bars and three press forged sheet bars from 3" diameter conditioned ingots prepared in the initial breakdown investigation were used for the initial rolling investigations. These six sheet bars were cut into two mults resulting in twelve pieces for rolling. Conditioning to remove cracks was necessary on the six press forged sections and grinding of these sections resulted in heat checks which were detrimental in the initial rolling operation. Three initial rolling temperatures of 2300°, 2500°, and 2700°F were used for breakdown rolling. Final rolling temperatures were 2100° and 1900°F. Four different reductions from the last recrystallization anneal were used. All pieces were to finish at a nominal .040" thick giving a total of 48 processing variables. In the initial rolling from sheet bar, no noticeable differences were observed due to the different rolling temperatures utilized. However, sandblasting after initial rolling revealed an erosion condition on the surface of the sheet which was progressively worse with the increase in rolling temperature. The as-rolled, press forged pieces exhibited severe crack propagation due to the heat checks present in the conditioned sheet bars. However, the as-rolled sections were conditioned to sound material and the initial rolling investigation continued in order to determine rolling parameters and generate mechanical property data. The intermediate rolling operations were accomplished at a 2300°F rolling temperature. The final rolling operations were accomplished using stainless steel cover plates in order to allow greater reduction per pass, to minimize heat loss, and to allow more efficient attainment of the final .040" gauge desired.

The final rolled sheet exhibited cracking tendencies due to both misalignment of the cover sheet material during rolling and handling of the material after the rolling operation. Laminations

were detected on the final rolled sheet along the extreme edges and ends of several of the sheets. However, no correlation could be made with the rolling practice. After shearing samples for mechanical and physical property determinations, delaminations were discovered on most of the sheared edges which extended from the sheared edge in to about .200" in depth.

From the bend transition and tensile data developed on the initial rolled sheet product, initial rolling temperature of 2300°F, final reduction from recrystallization of 92% to 95%, and a final stress relief anneal of 1800°F were optimum for the variables investigated.

InFab rolling of the two sheet bars produced in the initial breakdown investigation was accomplished at initial rolling temperatures of 3000° and 2400°F, respectively. Except for a coarser grain structure, due to the higher rolling temperature, the properties of the InFab rolled material were very similar to those of the conventionally rolled material. Surface condition of the InFab rolled material was excellent which reflected the high purity of the InFab atmosphere. However, due to this high purity and improved surface condition, bonding of the stainless steel cover plates to the tungsten sheet was experienced.

Six additional press forged sheet bars were applied for rolling to further refine the rolling parameters. The three major variables investigated in this series were cross rolling, final rolling temperature, and intermediate stress relief annealing. Based on minimum bend transition values and maximum tensile elongation on .040" sheet for initial rolling evaluation, the following optimum processing variables were established.

1. Initial rolling temperature - 2300°F furnace temperature.

2. Recrystallization anneal at 2500° to 2700°F.

3. Intermediate rolling at 2300°F furnace temperature.

4. Intermediate stress relief - one hour at 2000°F.

5. Final roll at 1250° to 1550°F.

6. Final stress relief - one hour at 1700°F

7. Reduction after last recrystallization anneal - 92% to 94%.

8. Reduction after stress relief anneal - 80% to 85%.

9. Cross rolling ratio 1:1, from last recrystallization anneal.

In attempts to scale-up to the 36" x 96" sheet requirements at gauges of .020", .040", and .060", problems were experienced in all phases of the processing sequence. A sheet size of .060" x 36" x 96" required a starting sheet bar size of 2" x 6" x approximately 32" long. This combination of thickness and length prevented the attainment of the initial percentage reductions required in the breakdown rolling operation. Due to the smaller percentage reduction, propagation of alligator type cracks were experienced (longitudinal cracking through the center of the sheet bar) and were directly related to the limiting mill capacity at this width and thickness. In order to acquire increased reduction on initial rolling of this sheet bar, the rolling direction was changed to the 6" width x 32" length. This resulted in increased reductions but lead edge and tail edge cracks persisted to develop causing an average 13% yield loss on all originally applied sheet bar.

Additional problems were experienced prior to the final rolling operation. Sheet sizes were as large as 30" x 30" and required cutting all edges and complete surface conditioning. These operations resulted in frequent cracking problems due to

handling and processing of this size and weight. After the final rolling operation, the sheets normally had edge cracks up to 1" resulting from stresses incurred from misalignment and slipping of the cover sheet material. In addition, the sheets were out of flat in the as-rolled condition. Hot shearing operations to remove the edge cracks and as-rolled ends frequently resulted in edge crack propagation due to the combination of the notch sensitive effect of existing cracks and the stresses incurred in shearing out of flat sheet. Attempts to flatten the sheet by roller leveling were made but some cracking was also experienced in this operation due to the notch sensitive effect of the edge tears and the necessity for handling the sheet in this condition. The necessity for handling the finish rolled and sheared sheets through stress relieving and finish inspection operations also resulted in occasional cracking situations.

D. General Observations

The problems associated with melting, extruding, and rolling with respect to surface tears, cracking, edge tears on sheet and overall handling prohibited the attainment of the contract objectives of 36" x 96" sheet in any of the three gauges under investigation. Mechanical properties of the finished production sheet were consistent with those values obtained from the initial rolling investigation. Recrystallization temperature for .020" gauge sheet was determined to be at 2300°F for 100% recrystallization and 2400°F for 100% recrystallization of the .040" and .060" gauge sheets. Bend transition temperatures range from 150° to 325°F on the longitudinal samples and 200° to 425°F on transverse samples. Ultimate tensile strength ranged from 90,000 psi to 120,000 psi in the longitudinal direction and 92,000 psi to 129,000 psi in the transverse direction. The 0.2% offset yield strength values ranged from 80,000 psi to 114,000 psi in the longitudinal direction and

from 80,000 psi to 116,000 psi in the transverse direction. Percent elongation at the 900°F test temperature averaged approximately 8% for all gauge samples. Three of the ultimate 21 sheets produced exhibited greater than 4% out of flat and the out of flat condition seemed directly proportional to the final width of the sheet.

The as-pickled surface condition of four of the sheets exhibited poor surface quality due to prior conditioning and oxide erosion. Gauge variation from sheet to sheet was generally within one-half of AMS 2242 tolerance. The final gauge of the sheets varied considerably due to problems in determining gauge during rolling caused by oxide build-up on the sheet and the use of cover plates to acquire the final reductions.

VI. Conclusions

1. Satisfactory tungsten ingots can be melted with consistent quality in small diameters up to 6" round.

2. Large diameter ingots (up to 9-1/2" round) can be melted providing adequate power and cooling capacity is available. However, erratic melting conditions resulted in poor sidewall and subsequent increased yield loss.

3. Occasional cracking of the 9-1/2" round ingot was experienced after a stress relief heat treatment due to thermal expansion and contraction.

4. Direct forging to sheet bars is not practical due to peripheral crack propagation.

5. Satisfactory extrusion of sheet bars and rounds from conditioned billet diameters up to 6" can be accomplished at extrusion ratios of 4.5:1 and 3000° to 3500°F furnace temperatures.

6. Press forging of extruded rounds to sheet bars can readily be accomplished but is not practical due to yield loss on conditioning and the addition of a processing step.

7. Extrusion of 8" diameter conditioned ingot to 2" x 6" sheet bar can be accomplished but surface tears in the extrusion could not be eliminated at the extrusion temperatures and ratios investigated.

8. The combination of minimum bend transition temperatures and maximum tensile properties were realized using rolling sequences as follows:

   a. Initial rolling temperature - 2300°F.

b. Recrystallization anneal - one hour at 2500° to 2700°F.

c. Intermediate rolling temperature - 2300°F.

d. Stress relieve one hour at 2000°F.

e. Final rolling temperature - 1400°F.

f. Reduction after recrystallization - 92% to 94%.

g. Reduction after stress relief anneal - 80% to 85%.

h. Cross rolling ratio - 1:1.

i. Final stress relief temperature - 1700°F.

9. InFab forging and rolling of extruded rounds resulted in identical properties to that of conventionally rolled material but better surface condition of the final rolled sheet was realized due to the InFab atmosphere.

10. The contract objectives of 36" x 96" sheet product at .020", .040", and .060" gauge were not realized due to severe problems with surface condition and cracking due to processing and handling in all processing sequences investigated.

VII. Recommendations

1. To improve melt characteristics and obtain better sidewall conditions in large diameter tungsten ingot melting, optimum conditions of purity, density, and concentricity of electrode material, combined with improved furnace design with respect to available power and cooling capacity is considered a necessity.

2. To eliminate surface tears resulting from the extrusion of large diameter tungsten billet to sheet bar, additional investigation with respect to grain size of the original ingot combined with evaluation of improved lubricating methods is recommended.

3. In order to successfully roll integral, high purity thin gauge tungsten sheet 36" x 96" the following recommendations are offered:

   a. Utilization of an adequate capacity four high hot rolling mill to insure maximum force for initial breakdown reductions and to attain final gauge on single sheets without the use of cover sheets.

   b. Incorporate heating and feed facilities to insure minimum heat loss during rolling.

   c. Design facilities so that rolling, shearing, and flattening can be accomplished while heat is maintained in the sheet.

   d. Design adequate handling devices so that support of the sheet is present in all necessary cold processing operations such as lifting, pickling, conditioning, heat treating, inspecting, and shipping.

APPENDIX I

SUMMARY OF THE STATE-OF-THE-ART ANALYSIS

DEVELOPMENT OF NEW OR IMPROVED TECHNIQUES FOR THE PRODUCTION OF TUNGSTEN SHEET

by

D. J. Maykuth, V. D. Barth, and H. R. Ogden

29 September 1960 - 29 December 1960

I. Introduction

This survey was conducted by the Battelle Memorial Institute with the assistance and cooperation of Universal-Cyclops. The objectives of the survey were to assess the current state-of-the art in the rolling of tungsten sheet and to recommend the composition(s) of a tungsten sheet material or materials for evaluation in the Phase II effort.

In conducting the survey, use was made of a questionaire and personal interviews as well as an extensive search of the literature and the Defense Metals Information Center.

The questionaire used is reproduced and included in this summary as Figures I-1 and I-2. This questionaire was mailed to approximately 140 organizations known or believed to have had experience in the production of tungsten or tungsten alloys or in converting compacts or ingots of these materials to sheet or other wrought forms.

Wherever possible, the data cited have been referenced to the pertinent Government reports or publications in which these have appeared. The opinions and recommendations given are the author's interpretation of the total information gathered during the survey.

Figure I-1

AMC TUNGSTEN SHEET ROLLING PROGRAM
[CONTRACT AF 33(600)-41719]
STATE-OF-THE-ART SURVEY

I. **Your Organization.**

A. **Are you a supplier of raw materials for use in producing tungsten?**

Yes____ No____

A producer of mill shapes? Yes____ No____

A consumer? Yes____ No____

Other interest: Research____; Alloy development____; Other____

B. Have you made tungsten products by:

Arc melting? Yes____ No____

Electron beam melting? Yes____ No____

Powder metallurgy techniques? Yes____ No____

Other procedures? Yes____ No____. If "yes", please state procedure used: ____

II. Raw Materials

A. What maximum levels of impurities of major importance are specified when procuring raw materials for use in the:

Consolidation of Tungsten by a Melting Procedure		Consolidation of Tungsten by a Powder Metallurgy Procedure	
________	________	________	________

B. What alloying and/or "doping" additions, if any, have been investigated?

1. Melting Procedure

Element or Compound	Quantity Added (or range of addition)	How Added
________	________	________

B-2. Powder Metallurgy Procedure

Element or Compound	Quantity Added (or range of addition)	How Added

C. What are the effects of alloying and "doping" additions noted in B (preceding page) on grain size, fabricability, and/or properties?

Element	Quantity Added (or range of additions)	Effect

D. Are you a supplier of tungsten raw materials? Yes____ No____

If yes:

Form	Size Range (for powders)

III. Consolidation.

A. Do you compact powder into shapes? Yes____ No____

If answer is "yes", what methods are used?

Compacting Method	Resulting Shape	Maximum Dimensions of Compacted Shape

B. What powder particle sizes and shapes are required for optimum compacting conditions?

Compacting Method	Average Particle Size	Particle Size Range	Particle Shape

C. What pressing procedures are used?

Pressure	Cold-Pressed Density	Lubricant Used (if any)	Die Shape

Additional comments on uniformity of density in cold-pressed shapes:

____

[Contract AF 33(600)-41719]

III. -D. What sintering procedures are used?

	For Electrodes	For Powder Metallurgy Process
Temperature		
Time		
Atmosphere		
Resulting as-sintered density		

E. What critical chemical reactions, if any, occur on sintering?

F. Where electrodes are produced for subsequent arc melting, how are sections joined?

G. What electrode-to-mold size ratios are used in arc melting?

H. What electrode configurations are used?

I. What are the optimum melting conditions according to your experience?

Electrode/mold ratio ____________________

Ingot size ____________________

Voltage ____________________

AC or DC ____________________

Amperage ____________________

Furnace atmosphere ____________________

Pressure above melt ____________________

J. Do you use any unusual processes in melting, differing from those commonly used in arc-melting practice? (mold liners, arc initiation, etc.)

[Contract AF 33(600)-41719]

III. -K. What is the maximum size of unalloyed tungsten arc-cast ingot which you can supply?

L. What is your normal yield on conditioned arc-melted ingots?

M. What are your electron-beam melting capabilities?

N. What methods are used for inspection of your consolidated product?

Inspection Method	Consolidated From Powder	Consolidated by Arc Melting
______	______	______

O. Please discuss any other means of consolidation not listed above, which you have employed:

IV. Fabrication.

A. Powder Metallurgy.

1. What are the maximum size wrought shapes you have made?

Forgings ______________________

Extrusions ______________________

Rolled bar ______________________

Sheet: gage	width	length
<0.020"	____	____
0.020"	____	____
0.040"	____	____
0.060"	____	____
>0.060"	____	____

IV.-B. Arc-Cast (or Otherwise Melted) Material.

1. What are the maximum size wrought shapes you have made?

Forgings ____________________

Extrusions ____________________

Rolled bar ____________________

Sheet: gage	width	length
<0.020"	______	______
0.020"	______	______
0.040"	______	______
0.060"	______	______
>0.060"	______	______

2. What conditions are used in initial breakdown for the production of sheet?

Method of Mechanical Working	Size of Workpiece	Preheating Tempera-ture	Preheating Atmos-phere	Lubricant (if any)
______	______	______	______	______

Extrusion ratio (if extrusion is used) ____________________

____

3. What reduction schedules are used in forging and rolling to sheet?

Working Operation	Preheating Temperature	Amount of Reduction Per Pass	Amount of Reduction Between Anneals	Annealing Tempera-ture	Annealing Atmos-phere
______	______	______	______	______	______

4. What is the size and separating force of your rolling mill?

____

____

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IV.-A-2. What conditions are used in initial breakdown for the production of sheet?

Method of Mechanical Working	Size of Workpiece	Preheating Temperature	Preheating Atmosphere	Lubricant (if any)

Extrusion ratio (if extrusion is used) ____________

3. What reduction schedules are used in forging and rolling to sheet?

Working Operation	Preheating Temperature	Amount of Reduction Per Pass	Amount of Reduction Between Anneals	Annealing Temperature	Annealing Atmosphere

4. What is the size and separating force of your rolling mill?

____

5. Do you use a protective atmosphere or protective coating?

Yes____ No____. Composition of atmosphere or coating,

if used: ____________

6. What surface conditioning treatments are used?

	Pickling	Grinding	Other
Intermediate			
Final			

7. What straightening or flattening procedures are used for final sheet?

Figure I-2

DATA SHEET FOR TUNGSTEN AND TUNGSTEN-BASE ALLOYS

Please include developmental alloys. For proprietary alloys please consider listing data without specifying alloy content.

Company ______________________________

Alloy Designation ______________________________

Composition ______________________________

This alloy is ____ **experimental** ______ **pilot plant** ________ **commercial.**

Method of Consolidation ______________________________

Initial Breakdown by ______________________________

Subsequent Fabrication by ______________________________

What special procedures or precautions are necessary in fabrication? ______________

____

Properties:

Recrystallization Temperature (specify amount of work and working temperature in piece tested.)

____

Tensile Properties

Tensile Data at Strain Rate of ______ in/in/min. in ______ atmosphere.

Condition	Test Temperature	Yield Strength (Kips)	Ultimate Tensile Strength (Kips)

% Elongation	% Reduction of Area	Elastic Modulus	Hardness

IV.-B-5. Do you use a protective atmosphere or protective coating?

Yes____ No____ Composition of atmosphere or coating, if

used: ____

____

6. What surface conditioning treatments are used?

	Pickling	Grinding	Other
Intermediate	______	______	____
Final	______	______	____

7. What straightening or flattening procedures are used for final sheet?

C. What are the significant variables in fabricating arc-cast vs. powder compacted shapes?

V. (a) What types of mechanical testing equipment do you have, and what are their limitations?

(b) What are your chemical analysis procedures and standards?

For carbon ____

____

For oxygen ____

____

For nitrogen ____

____

For metallics ____

____

DATA SHEET FOR TUNGSTEN AND TUNGSTEN-BASE ALLOYS

Creep and/or Stress Rupture Data:

Transition Temperature (specify type and testing conditions):

Oxidation Data:

Additional Comments:

Please attach reprints, if available, or references to published or inhouse reports or data sheets where these or additional property data on tungsten and tungsten alloys are available.

II. Summary

All tungsten sheet now being made commercially is prepared by powder-metallurgy techniques. In this process, tungsten powder with an average particle size in the range of 1 to 10 microns, is mechanically or isostatically pressed to sheet bar. Resulting bar densities of about 65% to 75% of theoretical are obtained. The bars must then be densified to a minimum of about 85% of theoretical by high-temperature sintering in hydrogen or vacuum. Sintering also has the desirable effect of purifying the powder, especially with regard to oxygen. The sintered product is then rolled directly to sheet, with or without the benefit of high temperature forging to further improve densification.

Most of the producers' effort with tungsten as a sheet material has been given to rolling the unalloyed metal. The maximum size sheet now available from United States producers is 17" wide x 17" long at 0.040" thickness (one producer). This same producer is currently engaged in a pilot development program, for the Bureau of Naval Weapons, which has the ultimate objective of producing 3500 pounds of 0.060" x 18" x 48" tungsten sheet by powder-metallurgical techniques.

Aside from unalloyed tungsten, only four tungsten sheet "alloys" have been produced on a commercial or semi-commercial basis. These include the 1% and 2% thoria alloys, available from several producers, and two doped grades, Types 218 and K-100, available from two individual producers. Each of these alloys is being made by powder-metallurgical techniques. Production of the thoriated grades is appreciably more difficult than for unalloyed tungsten. At the present time, these as well as the Type 218 grade are not available as sheet in widths above about 4". The K-100 grade has been rolled to sizes as large as 0.060" x 7" x 27" and 0.065" x 10" x 36".

At the present time, equipment limitations, rather than technological limitations, appear to be the major deterrent to increasing the size (width) capability of powder-metallurgy sheet. The need appears especially critical for protective-atmosphere furnaces capable of heating large-size sheet bars and slabs to the temperatures required for 1) adequate densification, and 2) preheating for breakdown rolling.

Satisfactory techniques have been developed for the extrusion and forging of sintered tungsten and tungsten-alloy billets. For satisfactory recoveries of material in these operations, sintered billet densities at least as high as those for direct rolling sheet bar are required. Neither extrusion nor forging is being used to convert massive, round sintered sections to sheet bar.

Experience in the consumable-electrode arc melting of tungsten is being accumulated rapidly. At least seven organizations have used this procedure to produce good quality unalloyed tungsten ingots in diameters of 4" or greater. Unalloyed tungsten ingots as large as 9" diameter have been made. So far as tungsten alloys are concerned, production melting capabilities have been demonstrated for only a few binary tungsten-molybdenum compositions, most notably the 85W-15Mo alloy which is now being melted by several producers in ingot diameters up to 12". Electron-beam melting of tungsten shows equally good promise for preparing tungsten ingots although present equipment is limited to a maximum tungsten-ingot diameter of 4".

Aside from two notable exceptions, no successes have been achieved in direct forging or rolling large-diameter (greater than 2") arc-cast tungsten or tungsten alloy ingots. In both of these instances, the key to success appears to lie with the use of grain-refining additions, and reproducibility of these successes

has not yet been demonstrated. Thus, at the present time, the most practical means of breaking down as-cast tungsten-alloy ingots is by extrusion.

Various facilities and organizations have successfully extruded arc-melted ingots of unalloyed tungsten and a variety of tungsten-base alloys. Most of this work has been done on an experimental basis. Nevertheless, sufficient progress has been made with unalloyed tungsten and a few tungsten-molybdenum alloys so that yields of better than 50% can be consistently expected in extruding these materials to simple rounds.

Through the use of extrusion, the feasibility of producing tungsten sheet from arc-cast material has been demonstrated. In this development work, sheet in sizes up to 0.040" x 6-1/2" x 17" was obtained from portions of extruded bar after subsequent forging to sheet bar and rolling at 2300°F. This experience indicates that breakdown rolling of arc-melted sheet bar (after breakdown by extrusion and forging) can be carried out at appreciably lower temperatures than those required for the sintered product (2700° to 2900°F).

The ductile-to-brittle transition temperature of tungsten has been shown to be sensitive to such variables as grain shape and size, strain rate, and metal purity. While the tensile transition temperature of tungsten sheet has not been determined, the ductile-to-brittle bend transition appears to occur over the same temperature interval found for that of tungsten bars and rods in tension, i.e. about 300° to 850°F. In all instances, the lower transition temperatures observed are for wrought or cold-worked material.

A fair amount of information has been generated concerning the effects of impurities on the properties of tungsten. Unfortunately, much of this remains qualitative in nature due to the

general inability of present analytical techniques to accurately measure impurity elements in tungsten at levels below about 10 ppm. Nevertheless, it has been shown that variations in the interstitial content of tungsten, in the range of 1 to 20 ppm, are apparently not a factor in determining the degree of low-temperature ductility in tungsten, i.e. at temperatures around the ductile-to-brittle transition. Rather, variations in trace metallic impurities are suspected. Similarly, interstitial impurities, in the ranges normal for sintered product, appear to have little effect on the recrystallization temperature of tungsten while variations in trace metallics can apparently affect the recrystallization temperature by as much as 700° to 900°F. With the improvement of analytical techniques, it is conceivable that effects of very low concentrations of interstitial impurities will be more evident.

The tensile strength of tungsten at temperatures through 2500°F appears quite sensitive to processing variables. The effect of these becomes less marked with increasing test temperature and, above about 3500°F, the tensile strength of tungsten appears essentially independent of both the consolidation practice used and prior thermal history. However, at temperatures above about 2500°F, the type of consolidation practice appears to have a marked effect on the degree of tensile ductility obtained. Thus, reduction-in-area values for sintered product decrease drastically above this temperature while high values are retained in arc-melted product at temperatures at least through 4200°F. Several organizations have shown that arc-melted ingot normally contains appreciably less impurities than sintered and fabricated product made from the same material. These purity differences appear to offer the main basis for explaining the high-temperature ductility differences between these types of material.

Several dilute tungsten-base alloys, prepared and tested as bar stock, show significant strength advantages over unalloyed tungsten at temperatures to about 3500°F. At higher temperatures, the only addition shown to improve the strength of tungsten is thoria, in amounts of 1% to 2%.

III. Conclusions and Recommendations

The following conclusions were reached as a result of this survey:

1. The only tungsten sheet materials which have a demonstrated production capability are unalloyed tungsten, the 1% and 2% thoria alloys, and two proprietary doped grades, i.e. Types 218 and K-100.

2. Each of these materials is being made by powder-metallurgical techniques which are essential, in all but the unalloyed grade, to obtain optimum properties through control of composition and dispersed particle size (i.e. thoria).

3. The largest size of unalloyed tungsten sheet made commercially is 0.040" x 17" x 17" (one producer) and this producer is currently engaged on Bureau of Naval Weapons Contract No. NOw 60-0621c which has, as the ultimate objective, the production of 0.060" x 18" x 48" tungsten sheet using powder-metallurgical techniques.

4. Thoriated tungsten sheet appears to offer the best prospects for obtaining a significant improvement in the strength of unalloyed tungsten at temperatures above about 3500°F, without substantially decreasing

*This conclusion is based on the assumption that strength properties obtained in thoriated tungsten bar stock can be obtained in thoriated tungsten sheet.

the recrystallization temperature of tungsten or substantially lowering its melting point. However, the preparation of large-sizes, thoriated tungsten sheet bars entails appreciably more difficulties than for unalloyed tungsten and the state-of-the-art for rolling wide thoriated tungsten sheets is considerably less advanced.

5. Of the two doped grades of tungsten being converted to sheet material, the greatest size capability has been demonstrated for the K-100 grade.

6. The feasibility of producing tungsten sheet from consumable-electrode arc-melted unalloyed ingot has been demonstrated by the Universal-Cyclops Specialty Steel Division. Through the use of extrusion to break down the cast structure and forging to sheet bar, pilot sheet samples have been obtained which compare favorably in size to the largest of those now being made using powder-metallurgy techniques.

7. No arc-melted tungsten alloys, in ingot sizes about 2" diameter, have been converted to sheet material.

8. Generally, arc-melted tungsten product is characterized by a higher total purity than can be presently obtained by powder-metallurgy consolidation practices. The higher purity associated with the arc-melted product may be expected to contribute to greater ductility in this material at elevated temperatures. This has already been reflected in the successful use of lower rolling temperatures for arc-melted product (after extrusion and forging). Conversely, the higher purity may lead to a reduction in the recrystallization temperature.

9. It is recognized that, in the ultimate scale-up of a rolling practice for arc-melted billet, some difficulty may be encountered by the lack of adequate heating facilities for extruding the large billets required.

On the basis of these conclusions, the following recommendations are made:

1. It is felt that the presently active Air Force sheet rolling program should be confined to unalloyed tungsten sheet product, fabricated from arc-melted material. One reason for this stand is that the Bureau of Naval Weapons tungsten sheet rolling program, being conducted by Fansteel Metallurgical Corporation, is thoroughly investigating the powder-metallurgy approach to the consolidation and fabrication of sheet from unalloyed tungsten and variously doped tungsten powders. It, therefore, is thought needless to duplicate that effort, as the results of that work program will be available for comparison with the results of work performed to produce sheet from arc-cast material.

2. It is recognized that somewhat higher strength at temperature, than available in tungsten, may be required for certain structural applications and some types of rocket nozzles. According to present knowledge, thoriated tungsten, a powder product, is the best example of such an alloy. However, the preparation of thoriated tungsten sheet bars entails appreciably more difficulties than for unalloyed tungsten. At such time as the Bureau of Naval Weapons program develops optimum techniques for producing

powder-compacted sheet bars that can be fabricated to high-quality sheet, it may be a logical follow-up for an Air Force program to provide wide, powder-metallurgy, thoriated tungsten sheet.

3. It is thought that by confining the AMC sheet rolling program to arc-cast unalloyed tungsten material, the state-of-the-art will be more satisfactorily and rapidly advanced than by having the effort divided between a powder-metallurgy approach and an arc-casting process. To provide the sheet size requirements of the AMC program will require ingot sizes considerably larger than those heretofore converted to sheet material. This will introduce new problems in melting and primary breakdown. Also, there will be need to investigate the effect of numerous processing variables on the purity, mechanical, and physical quality of the sheet product. The objective of the program is not only to produce a given size sheet of minimum dimensional variation, and free of laminations and other defects, but to develop processes which will provide chemical and structural homogeneity, the lowest possible ductile-to-brittle transition temperature and consistency of recrystallization behavior. If the aforementioned industry capability is accomplished by the AMC project, for unalloyed arc-cast tungsten, it will be possible at some future date to readily accomplish sheet production of arc-melted tungsten alloys indicated suitable from laboratory alloy-development programs.

4. It is strongly recommended that unalloyed arc-cast tungsten be the material from which sheet is to be fabricated by the subject project.

[illegible] II

Specification [illegible] 61-3-A
Date - March 21, 1961

REFRACTOMET DIVISION
UNIVERSAL-CYCLOPS STEEL CORPORATION
BRIDGEVILLE, PENNSYLVANIA

MATERIAL SPECIFICATION

TUNGSTEN ELECTRODE BARS

CONTENTS

SCOPE

This specification covers isostatically pressed and hydrogen [illegible] or vacuum sintered, high purity tungsten and [illegible] alloy powder electrode bars for consumable electrode [illegible] arc melting.

[illegible]

Electrode bars furnished to this specification shall be manufactured from metal hydrogen reduced powder produced by the [illegible]tate process. Other processes will be subject to [illegible] evaluation prior to approval. All electrode bars [illegible] for a given order shall be made from not more than [illegible] lots unless a deviation is provided in writing by [illegible] authorized representative of the Refractomet Division, [illegible]-Cyclops Steel Corporation.

CHEMICAL COMPOSITION

Electrode bars shall conform to the following maximum limits. Deviations for alloying requirements will be specified on the purchase order.

Element	Maximum Percent By Weight	Element	Maximum Percent By Weight
As	0.001	*Co	0.001
Al	0.001	Cu	0.001
Mg	0.001	Mo	0.005
Ca	0.003	Mn	0.001
Na	0.003	Sn	0.001
K	0.003	*Pb	0.001
Si	0.001	C	0.0025
Fe	0.002	$*O_2$	0.010
Cr	0.001	$*N_2$	0.0025
Ni	0.001	$*H_2$	0.0025

* - Reported only when specified on purchase order. Deviation for gas analysis will not represent cause for rejection.

The minimum tungsten content as measured by difference shall be 99.950 percent for unalloyed electrodes.

CHEMICAL ANALYSIS

4.1 Analysis of metallic impurities will be measured by spectrographic methods and may be taken on either the powder or electrode bar.

4.2 Carbon will be measured by the Leco Conductometric method on a sample of the pressed and sintered metal.

4.3 All gas analysis will be measured on a sample of the pressed and sintered metal utilizing inert or vacuum gas fusion techniques.

4.4 Check analysis by an outside source will be used when the chemistry limits as determined by Universal-Cyclops do not meet those specified in Paragraph 3.1 of this specification. The results of this check analysis will be below the follow limits:

Element	Maximum Percent By Weight	Element	Maximum Percent By Weight
As	0.002	Ni	0.002
Al	0.002	Co	0.001
Mg	0.001	Cu	0.001
Ca	0.003	Mo	0.005
Na	0.003	Mn	0.001
K	0.003	Sn	0.001
Si	0.002	Pb	0.002
Fe	0.003	C	0.005
Cr	0.002		

Deviation from gas analysis will not represent cause for rejection.

5. PHYSICAL REQUIREMENTS

5.1 Apparent density, measured by dividing billet weight by billet volume expressed as a percentage of the theoretical density (19.3 gms/cc), may be reported but is subject to meeting the following density check by Universal-Cyclops on a qualifying basis.

The density of all electrode bars must not be below 90% of theoretical. On bars over two inches in diameter, the density will be measured within the center one inch diameter at a distance from the end at least equal to the diameter of the bar to be tested.

5.2 The nominal diameter shall have a tolerance as follows:

1" - 2.9" diameter $\pm$ 3/32"
3" - 7" diameter $\mp$ 1/8"

5.3 Local variations not exceeding 2" in length will not deviate from the nominal diameter by more than the following:

1" - 2.9" diameter	+ 3/32"	- 1/8"
3" - 7" diameter	+ 1/8"	- 1/4"

5.4 The difference between the major and minor diameters will not exceed the following:

1" - 2.9" diameter	3/16"
3" - 7" diameter	1/4"

5.5 Bars up to 2" in diameter will have flat ends perpendicular to within 1/8" of the longitudinal axes of the electrode. Bars greater than 2" in diameter will be perpendicular to within 1/4".

5.6 Electrode bar lengths shall be as follows unless otherwise mutually agreed to between vendor and purchaser.

Diameter	Minimum Lengths
1" to 1-1/2"	24"
Over 1-1/2" to 2"	30"
Over 2" to 3"	35"
Over 3"	45"

6. QUALITY

6.1 All electrodes supplied to this specification must be free of absorbed moisture, stains of foreign material, cracks, laps, seams, and surface oxidation.

6.2 This material should be satisfactory for consumable electrode vacuum arc melting processes and shall not exhibit spalling or evidence other undesirable characteristics which are detrimental to this process.

7. PACKING

All electrode bars supplied to this specification must be packed as follows:

7.1 Bars must be individually identified by powder lot and bar number, and packed in plastic bags containing a porous packaged desiccant and properly packed to prevent injury in shipment.

7.2 A packing slip should be furnished both inside and outside of each box and shall contain the following information:

a. Purchase Order Number.
b. Date of Shipment.
c. Government Contract Number, if supplied and requested.
d. Individual Bar Identifications, Weights, Dimensions, and Lot Numbers.
e. A statement as follows: "All material is being supplied in accordance with Universal-Cyclops' Tungsten Electrode Specification WEB 61-3-A." Deviations as specified on purchase order or by separate written approval must be indicated.

8. TEST REPORTS

8.1 The vendor will certify chemical analysis as indicated in Section 3.1 of this specification. A certificate on each lot of material supplied to this specification giving the results of the analysis must be packed with the electrodes. In addition, two copies will be forwarded by letter.

8.2 In addition to chemistry, the test report must contain the following information:

a. Purchase Order Number
b. Powder Lot Number
c. Specification Number
d. Nominal Electrode Diameter
e. Certification that material has passed the requirements of this specification.

9. REJECTION

Any purchase order which carries this specification number shall constitute an agreement whereby material which does not conform to this specification shall be subject to immediate rejection at the discretion of the purchaser. The only exceptions to this provision of the specification shall be those formally processed with the order acknowledgement and accepted as a deviation by the purchaser.

APPENDIX III

DESCRIPTION OF FACILITIES

The following is a brief description of the equipment referred to in the text which was utilized to process materials under this contract.

KD Furnace

A cold mold, vacuum consumable electrode, arc-melting furnace with an available DC power supply of 25,000 amps and equipped with a KB1200 pumping system. It is capable of producing ingots from 4" diameter to 13" diameter weighing up to 3000 pounds.

KC Furnace

A laboratory size furnace similar to KD furnace capable of melting 3" to 6" diameter ingots with 6,500 amps power supply.

700 Ton Loewy Extrusion Press (TAPCO Division, TRW)

Container size - 3.125" diameter
Maximum speed - 30" IPS (no load conditions)
23" IPS (load of 100,000 psi)
Billet heating - Inert atmosphere induction furnace
30KW motor generator - 4200 CPS
Temperature measurement - PT/Pt-Rd to 3000°F
W/W-Re above 3000°F
Extrusion Record - Brush Oscilloscope records pressure, speed, and ram travel
Dies - Ceramic coated die steel

2750 Ton Extrusion Press (E. I. duPont deNemours - Baltimore)

Maximum container size - 8" diameter
Billet heating - Argon inert atmosphere induction furnace

Temperature measurement - Shaw meter and optical pyrometer

Billet lubrication - Powdered 3 KBA glass coating

InFab

An inert atmosphere fabrication facility built by Universal-Cyclops under Contract NOa 55-006-c designed to forge and roll refractory metals at temperatures up to 4000°F. Atmosphere and equipment statistics are shown with layout in Figure III-1.

Hydrogen Annealing Furnace

A General Electric box furnace with a 17-3/4" x 40" x 108" hot zone utilizing molybdenum elements and an atmosphere of flowing hydrogen capable of heating to 2800°F. A laboratory furnace of similar type capable of reaching 3100°F with a hydrogen cooling chamber attached is also available. A gas fired preheat furnace capable of 1500°F is adjacently located.

Straightening Press

A 1500 ton vertical hydropress with a 36" square ram and a stroke of 8" used in conjunction with the hydrogen box furnaces.

Ultrasonic Units

A Sperry UR600 Reflectoscope with a RA recording attachment operating in an immersion tank 3' deep and 15' long with an automatic positioner and rotator. Also a Sperry UR500 portable unit is used for contact testing.

Forging Press

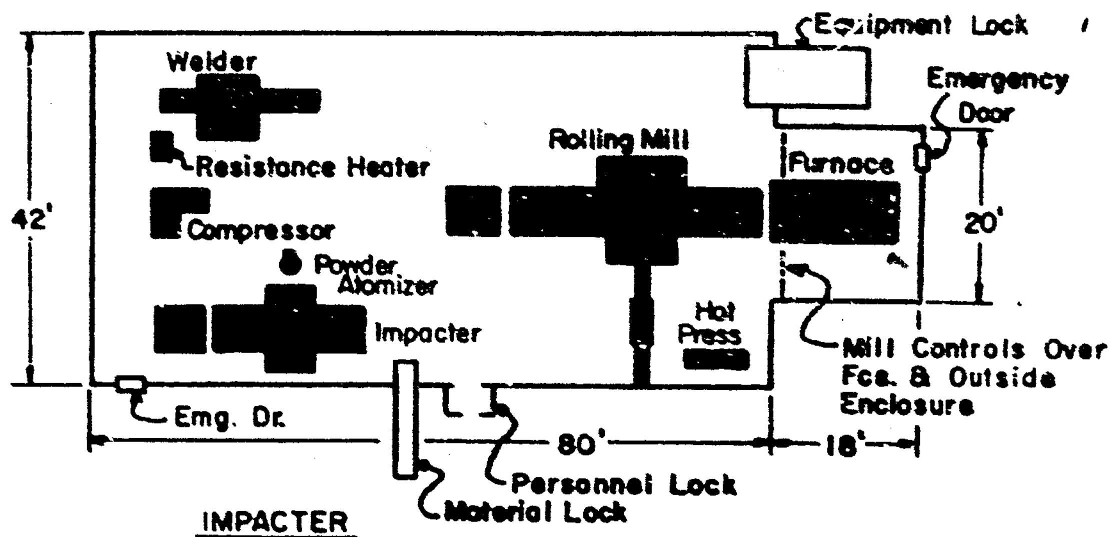
A 2000 ton vertical Loewy fast acting hydropress with a 48" wide x 92" opening used in conjunction with a 5 ton manipulator and a 5 ton charging machine for the eight gas fired furnaces available. This press is located at the Titusville Plant of Universal-Cyclops Specialty Steel Division.

ATMOSPHERE STATISTICS

ARGON 99.998

OTHER .002 — OXYGEN - .0002; NITROGEN - .0010; HYDROGEN - .0001; WATER VAPOR - .0002; HYDRO CARBONS - .0002

RECIRCULATION RATE (GAS) 36,000 CFH



ROLLING MILL

ROLL DIMENSIONS - 14" & 16" X 36"
SEPARATING FORCE - 850,000 LB
SPEED - 230 FT. PER MIN.
TEMPERATURES - 4000°F

IMPACTER

DIE DIMENSIONS - 7.5" X 15"
ENERGY OF IMPACT - 15,000 FT. LBS.
TEMPERATURE - 4500°F

PERSONNEL SUITS

TEMPERATURE - 70°F
AIR FLOW >700 CFH
REFRIGERATED DRY AIR

WELDER

36" STAKE SEAMER
POWER SUPPLY - 300 AMPS
ROTATING POSITIONER - 500 LB. CAPACITY
HEAD - AUTOMATIC TIG & MIG

HOT PRESS

150 TON CAPACITY

POWDER ATOMIZER

100 LB. FURNACE

ENCLOSURE STATISTICS

VOLUME - 84,032 CU. FT.
PRESSURE - .5 OZ. ABOVE ATMOSPHERE
TEMPERATURE - 70 - 120°F
POWER - 30,000 KWH/DAY
WATER - 743 GPM MAX. WHILE FORGING

EQUIPMENT LOCK

DIMENSIONS - 8' x 8' x 16'

FIGURE III-1
INFAB SPECIFICATIONS

Roughing Mill

A 2 Hi mill with 26" diameter x 48" face rolls driven by a 600 HP, 293 RPM Allis-Chalmers AC motor. This and the slow fire mill are located at Universal-Cyclops Pittsburgh Plant.

Finishing Mill

A 2 Hi mill with a 26" diameter x 32" to 56" face rolls on the same drive as the roughing mill. Gas fired one and two chamber furnaces are adjacent to both mills.

Roller Leveler

A 4 Hi Unger leveler for flattening sheet at Pittsburgh Plant.

Tensile Machine

A universal testing machine of 0 to 50,000 pounds capacity at variable head speed of .025" per minute to 8" per minute. Used for tensile and bend testing below 900°F in air. For test temperatures above 900°F, an adapted arc-weld stress rupture machine is used in conjunction with a vacuum furnace.

Hardness Tester

A Vickers diamond pyramid hardness testing machine using 1 KG to 150 KG loads.

Microscopes

Three bench microscopes ranging in magnification from 9X to 1500X.

Metallographs

A. O. 50X to 1500X with polarized light and phase I.D. apparatus. A B&L 25X to 2000X with polarized light half aperture and dark field illumination.

Polishers

DISA electro-polished and etched - 1.5A and 150V capacity. Bueller electro-polisher and etched - 10A and 100V capacity. Cintron automatic polishers for 1-1/4" diameter mounts. Bueller three wheel, two speed polish table, Bueller two wheel variable speed polish table.

Mounting Press

Bueller speed mounting press for 1-1/4" diameter mounts.

Cameras

Kodak 8" x 10" view camera. Graphic 4" x 5" camera. Omega 5" x 7" and larger.

Washer and Drier

Pako print washer, Pako continuous drum print drier, Fisher negative drier.

APPENDIX IV

SHEET ROLLING EVALUATION
SUMMARY OF HARDNESS ANNEALING CURVES

I. Introduction

In Table XXII is outlined the summary of sheet bar application for the sheet rolling evaluation. This schedule indicates two different type sheet bars in the recrystallized as well as the as-extruded condition. Initial rolling temperatures of 2300°, 2500°, and 2700°F were employed. Intermediate rolling temperatures of 2300°F were employed while final rolling temperatures ranged from 1900° to 2100°F. The percent reduction in area varied from 40% to 99%.

II. Summary

The fourteen curves shown in this appendix elucidate the behavior that can be expected from the variables employed. These curves show that the initial rolling temperature has little effect on the resulting hardness in the as-rolled condition or after heat treatment. Conversely, the curves show that the as-rolled hardness and recrystallization rate increase with increasing reductions.

For more specific information on the rolling parameters or other evaluations, the text should be consulted.

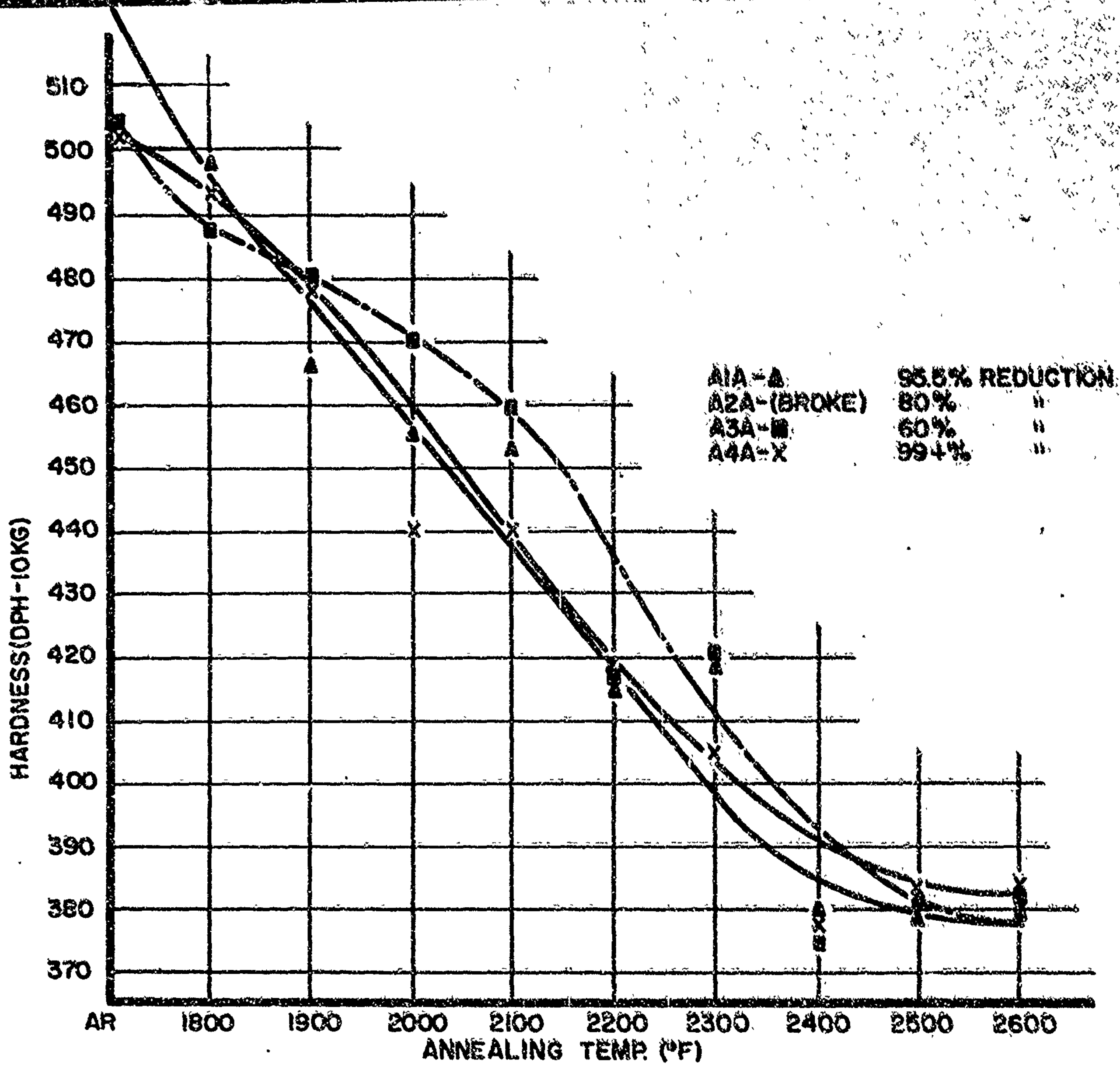


FIGURE IV - I
EFFECT OF REDUCTION ON THE RESPONSE TO HEAT TREATMENT - 0.040" SHEET

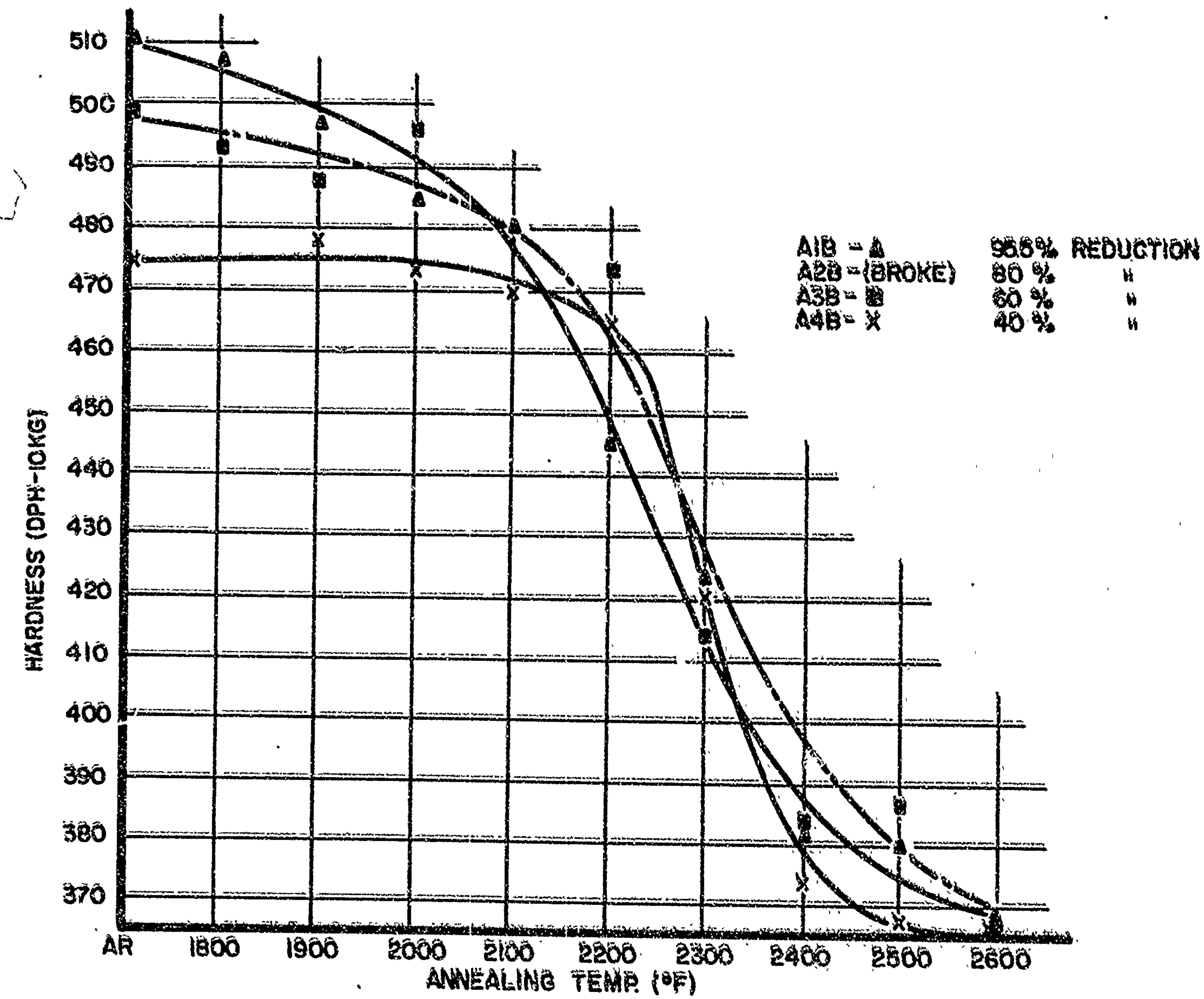


FIGURE IV-2
EFFECT OF REDUCTION ON THE RESPONSE TO HEAT TREATMENT ".0040" SHEET

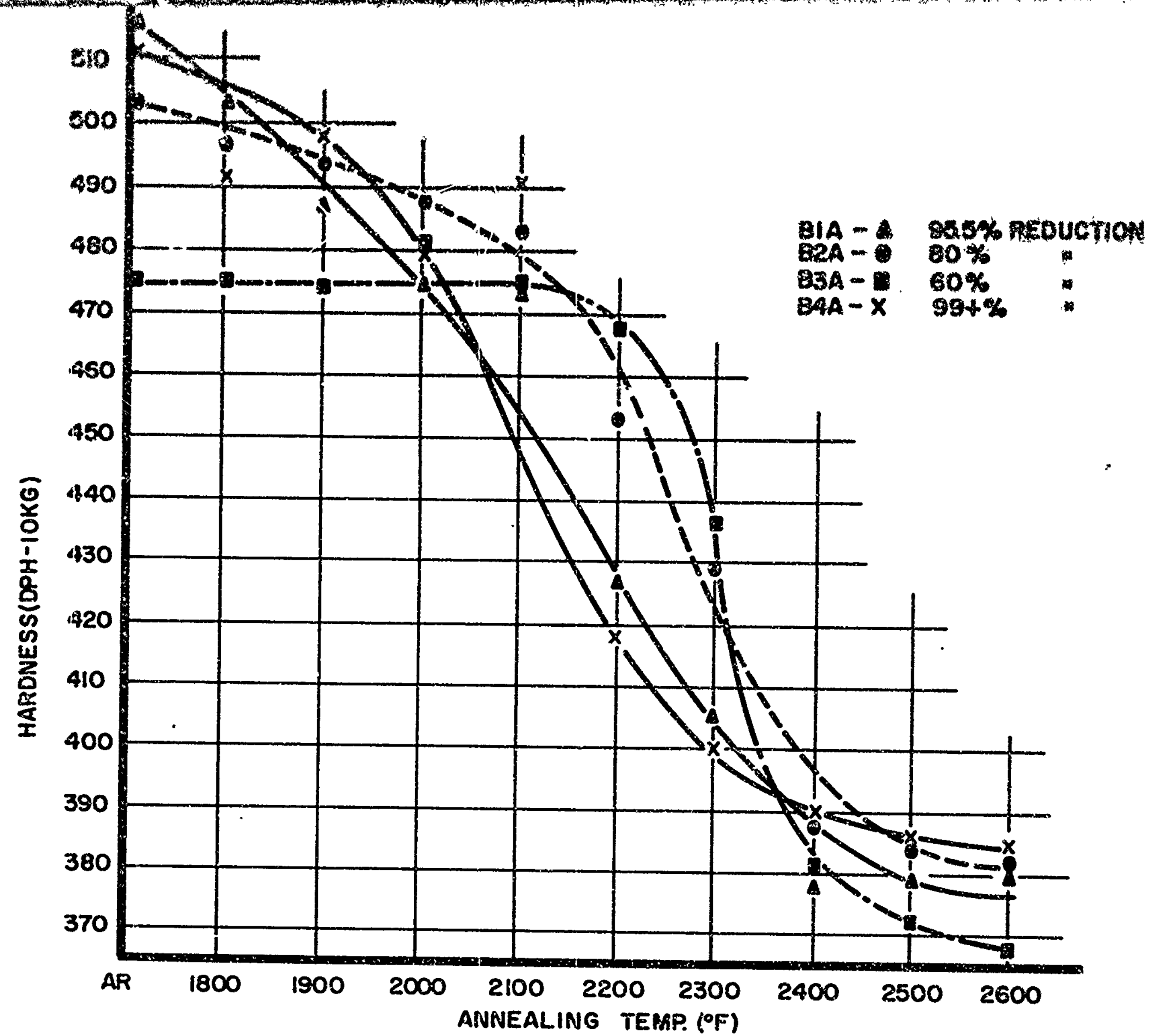


FIGURE IV-3
EFFECT OF REDUCTION ON THE RESPONSE TO HEAT TREATMENT-0.040" SHEET

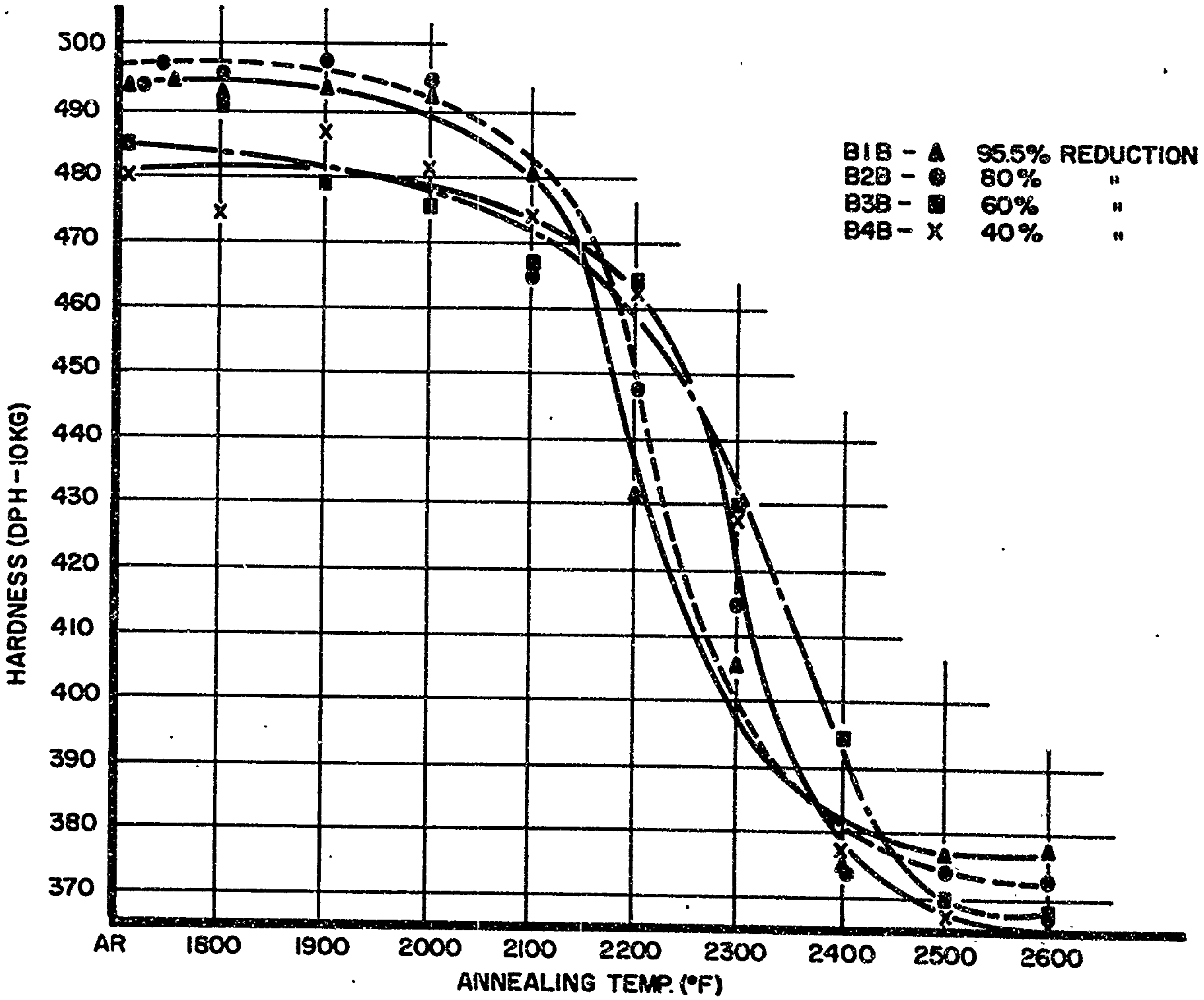


FIGURE IV-4

EFFECT OF REDUCTION ON THE RESPONSE TO HEAT TREATMENT -0.040" SHEET

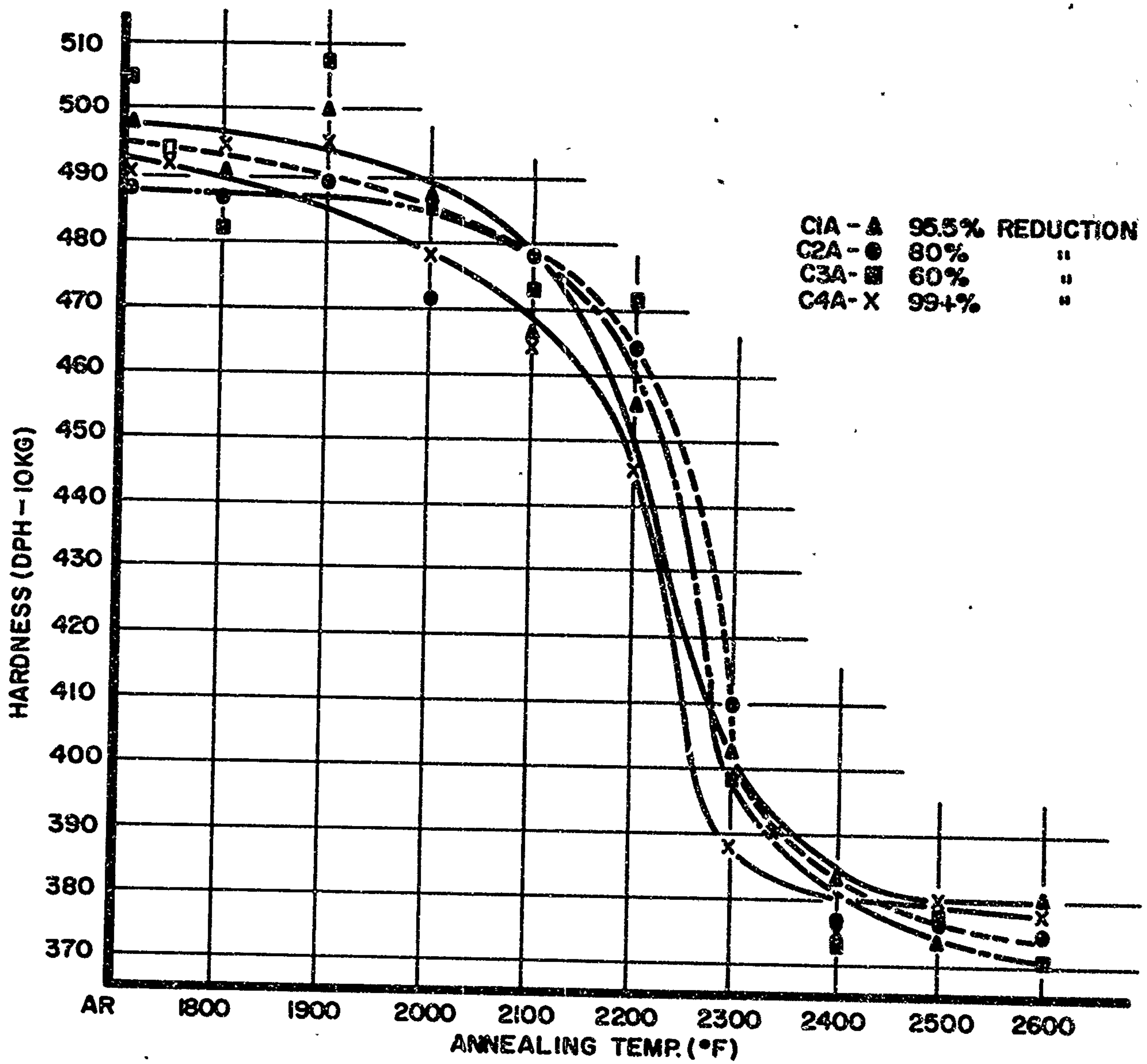


FIGURE IV-5
EFFECT OF REDUCTION ON THE RESPONSE TO HEAT TREATMENT-0.040" SHEET

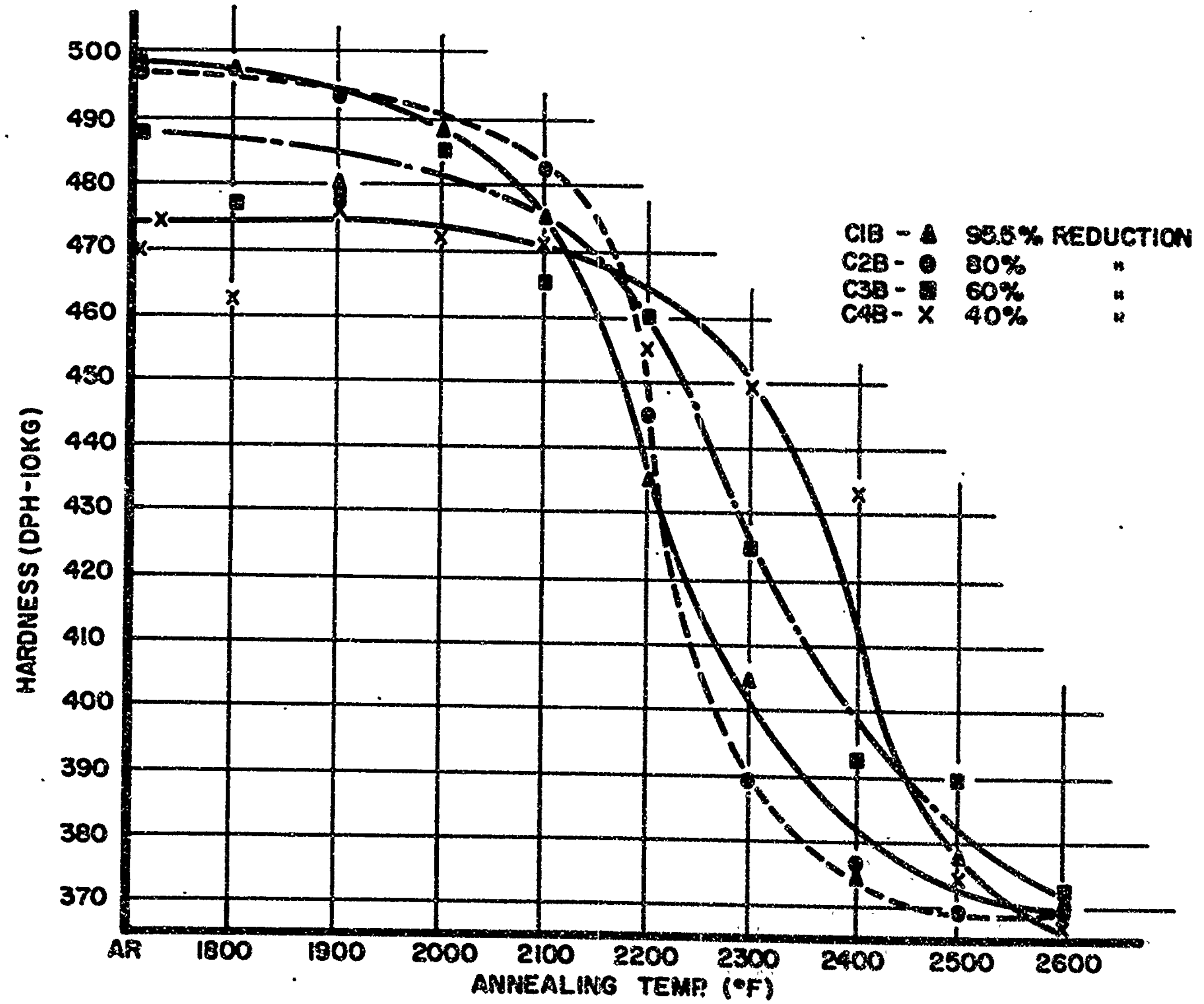


FIGURE IV-6
EFFECT OF REDUCTION ON THE RESPONSE TO HEAT TREATMENT - 0.040" SHEET

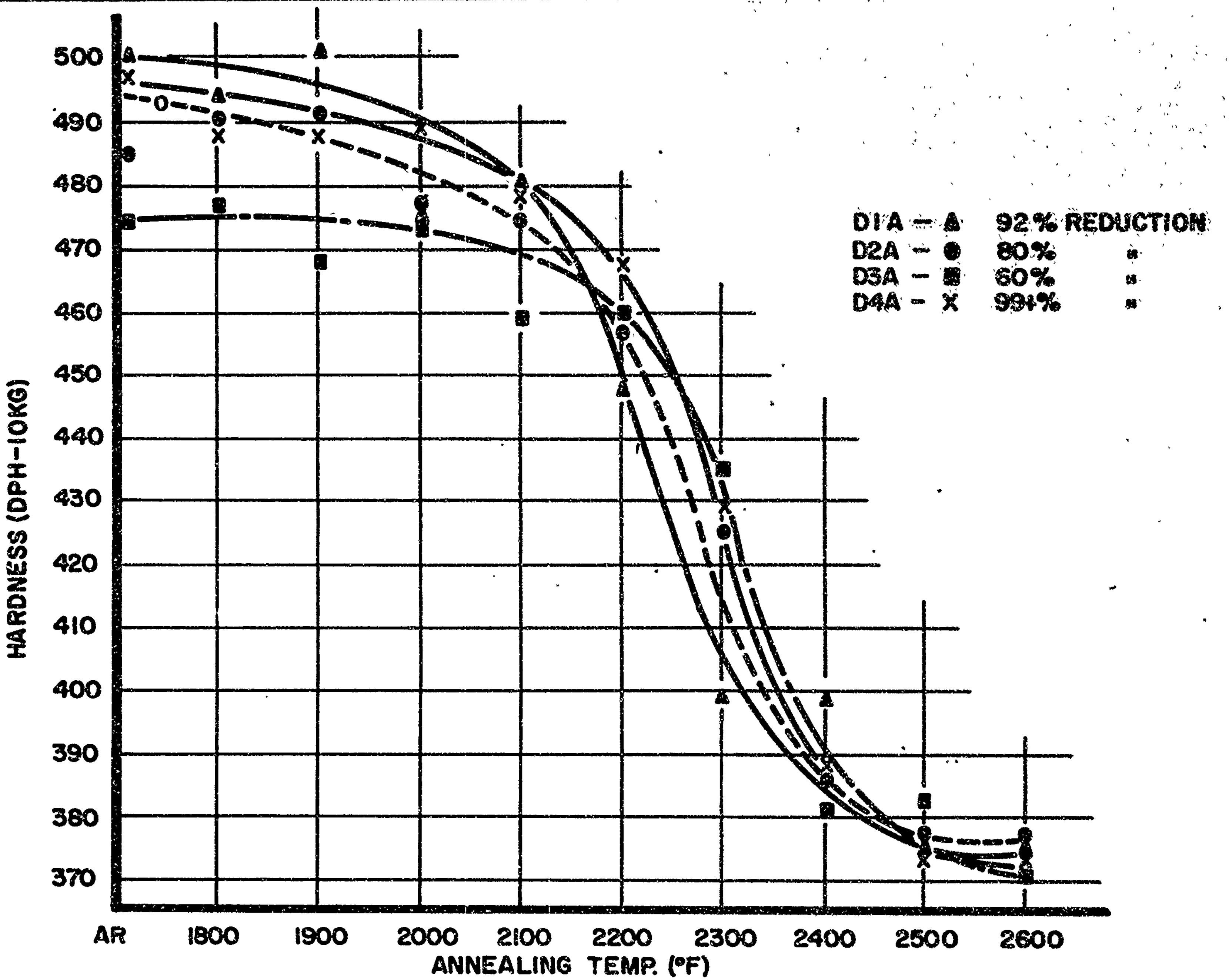


FIGURE IV-7
EFFECT OF REDUCTION ON THE RESPONSE TO HEAT TREATMENT - 0.040" SHEET

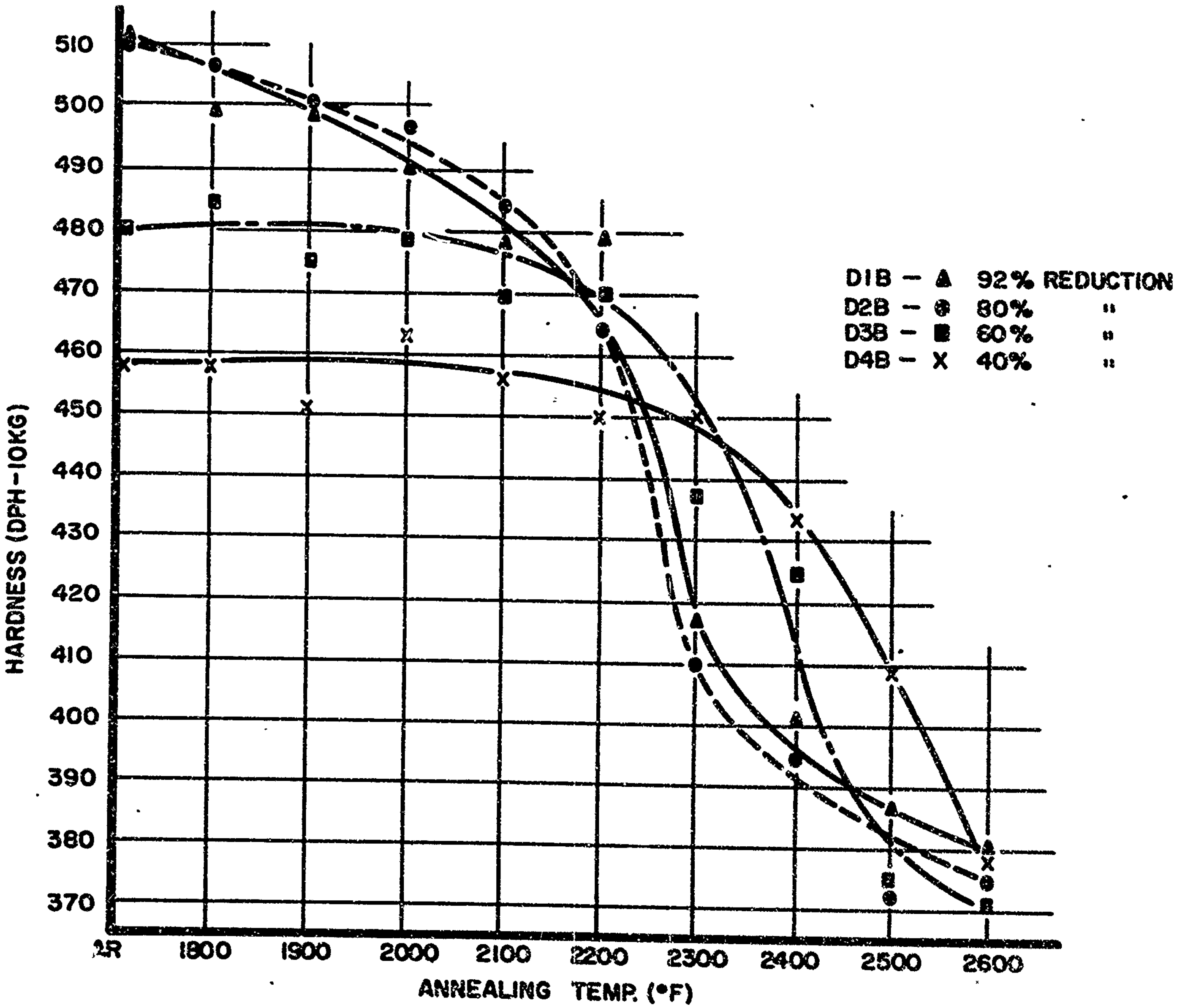


FIGURE IV-8

EFFECT OF REDUCTION ON THE RESPONSE TO HEAT TREATMENT - 0.040" SHEET

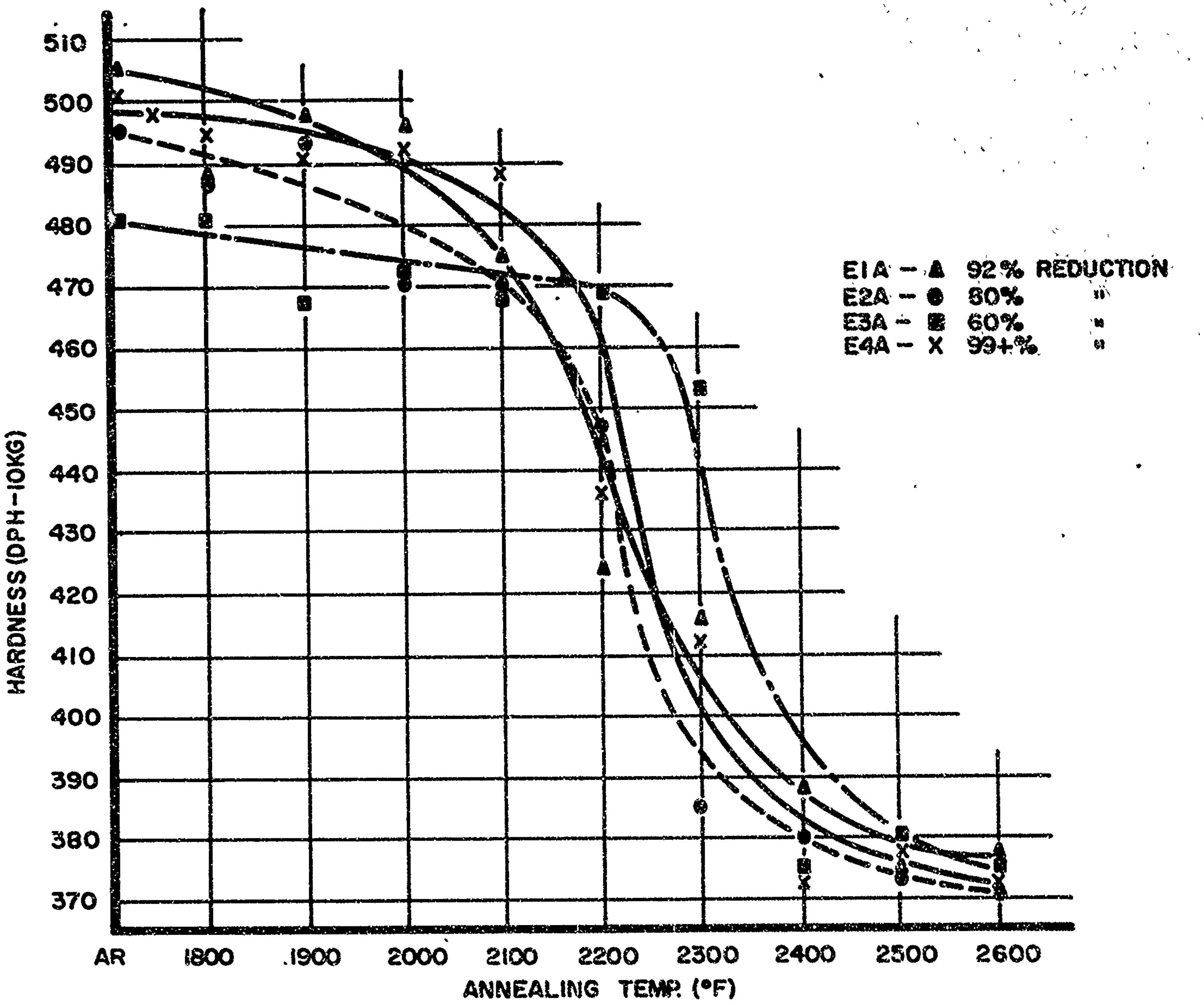


FIGURE IV-9
EFFECT OF REDUCTION ON THE RESPONSE TO HEAT TREATMENT - 0.040" SHEET

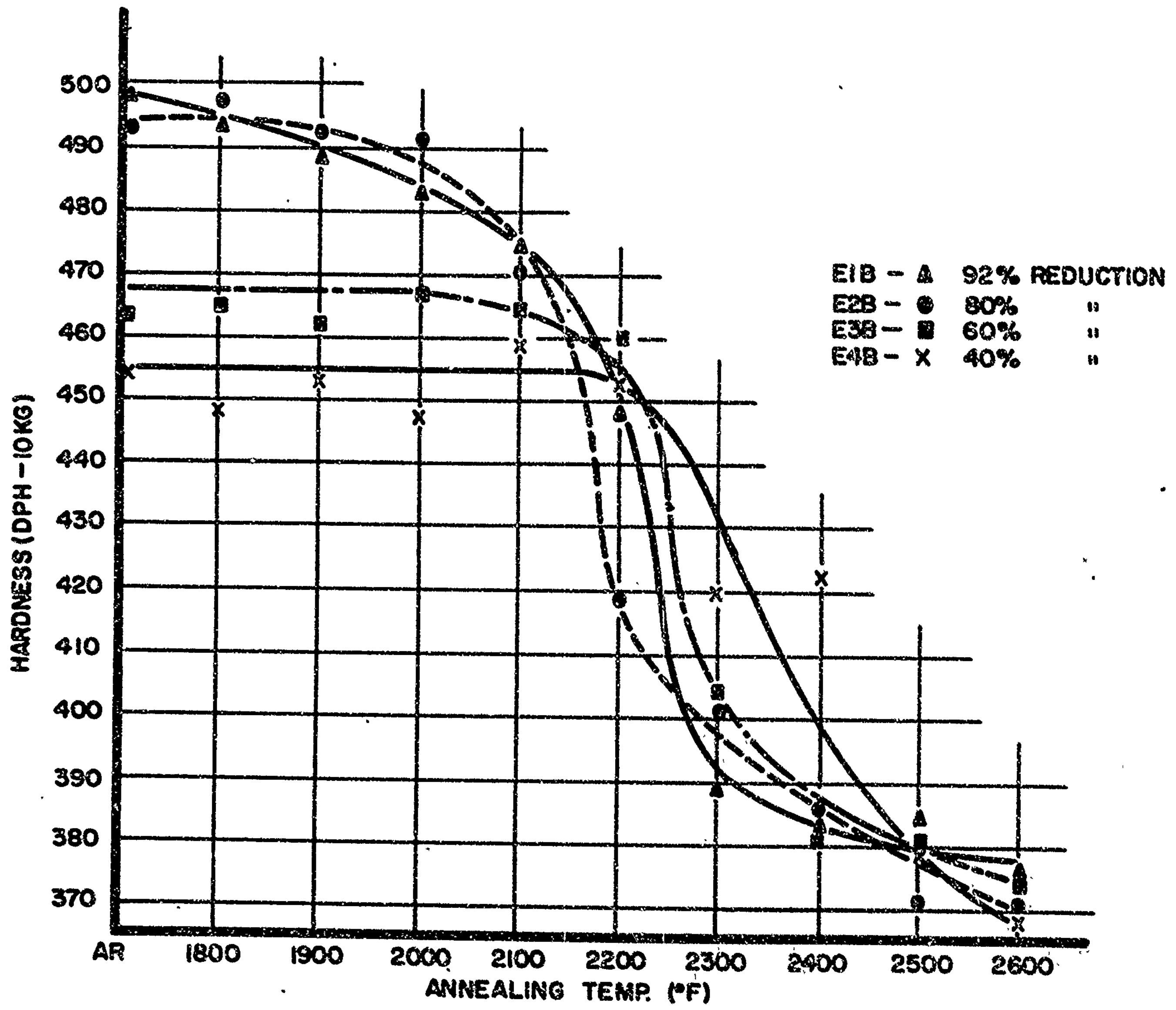


FIGURE IV-10
EFFECT OF REDUCTION ON THE RESPONSE TO HEAT TREATMENT – 0.040" SHEET

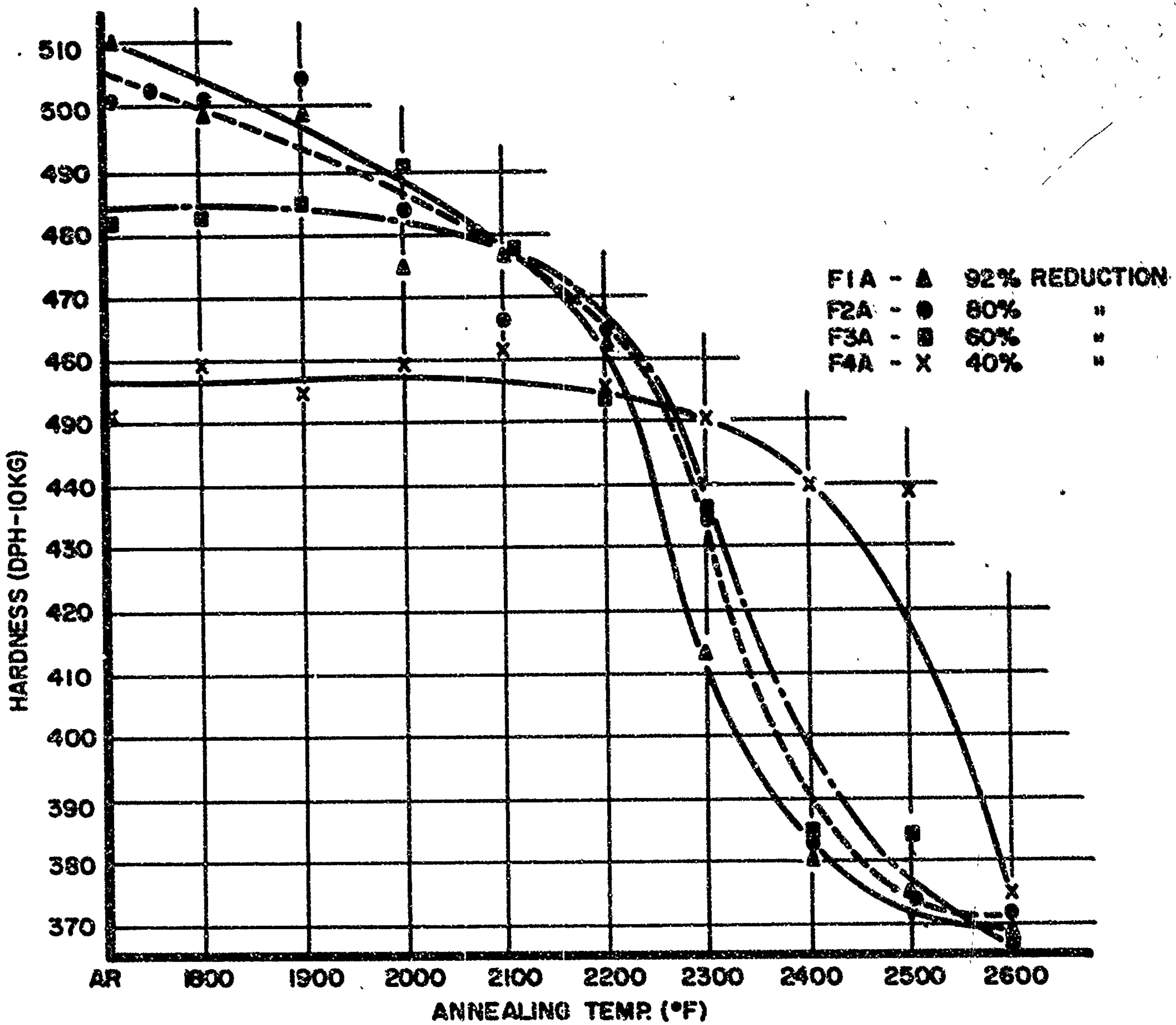


FIGURE IV-11

EFFECT OF REDUCTION ON THE RESPONSE TO HEAT TREATMENT - 0.040" SHEET

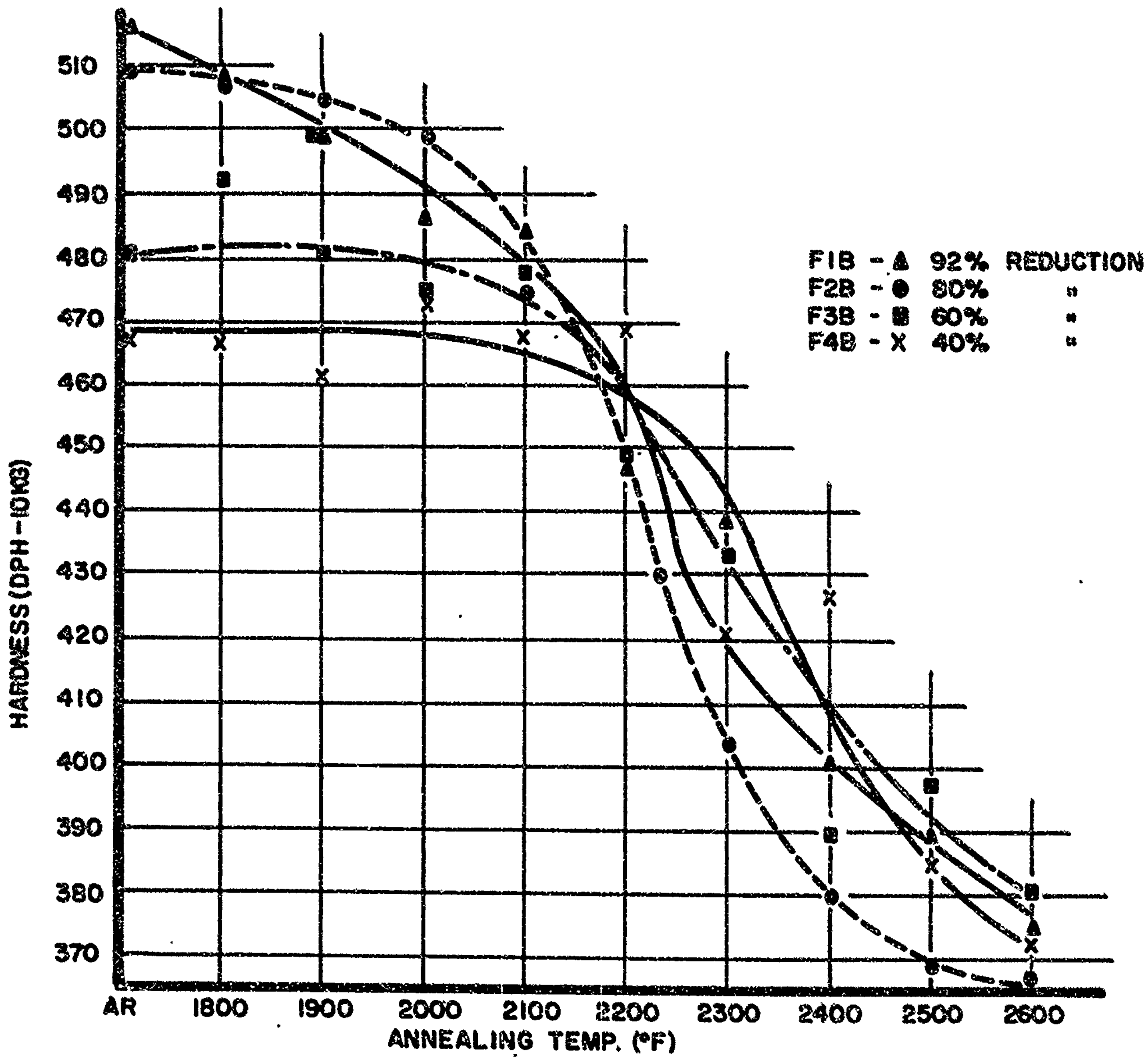


FIGURE IV-12

EFFECT OF REDUCTION ON THE RESPONSE TO HEAT TREATMENT - 0.040" SHEET

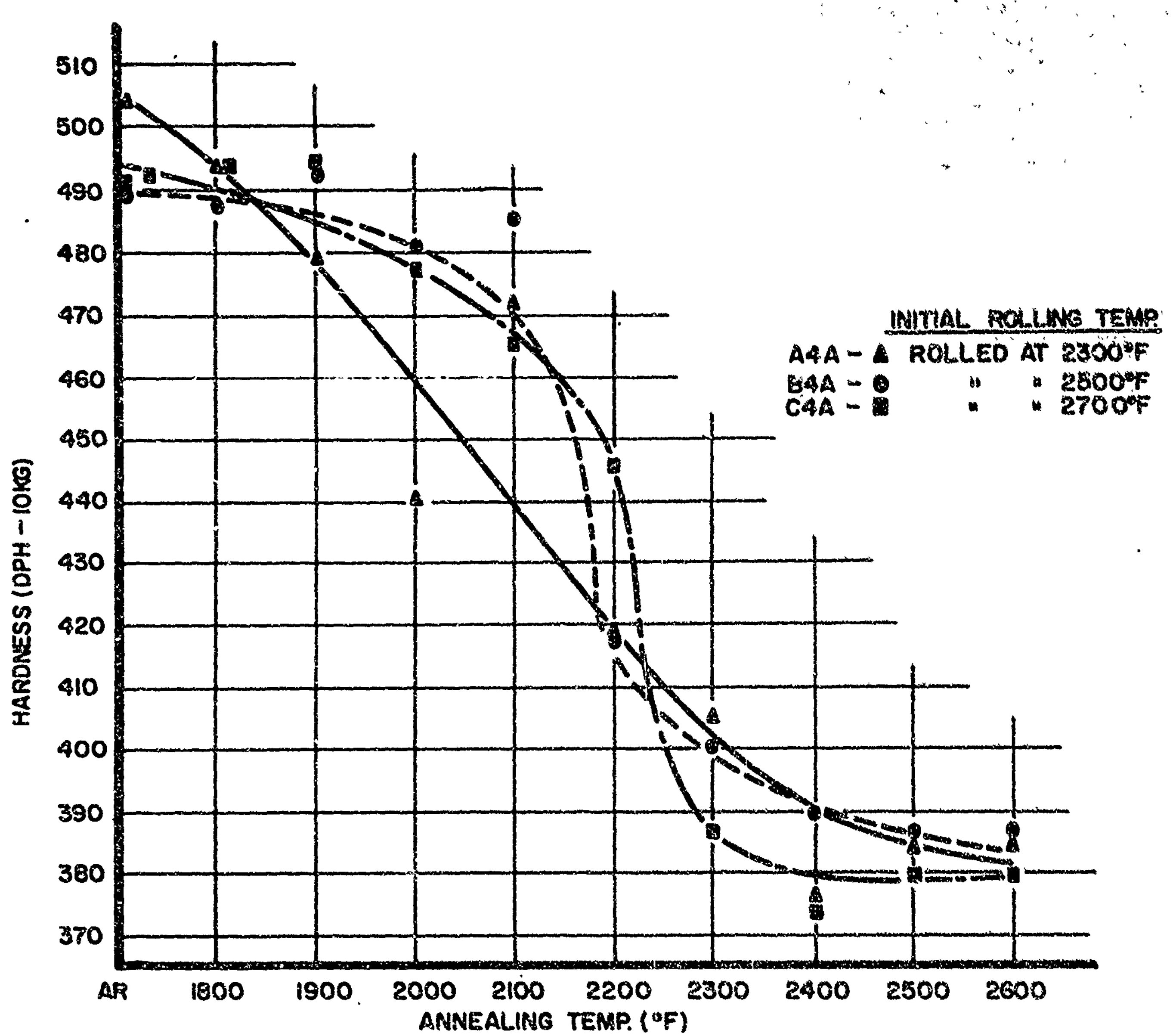


FIGURE IV-13
EFFECT OF INITIAL ROLLING TEMPERATURE ON THE RESPONSE TO HEAT TREATMENT (CONSTANT REDUCTION)

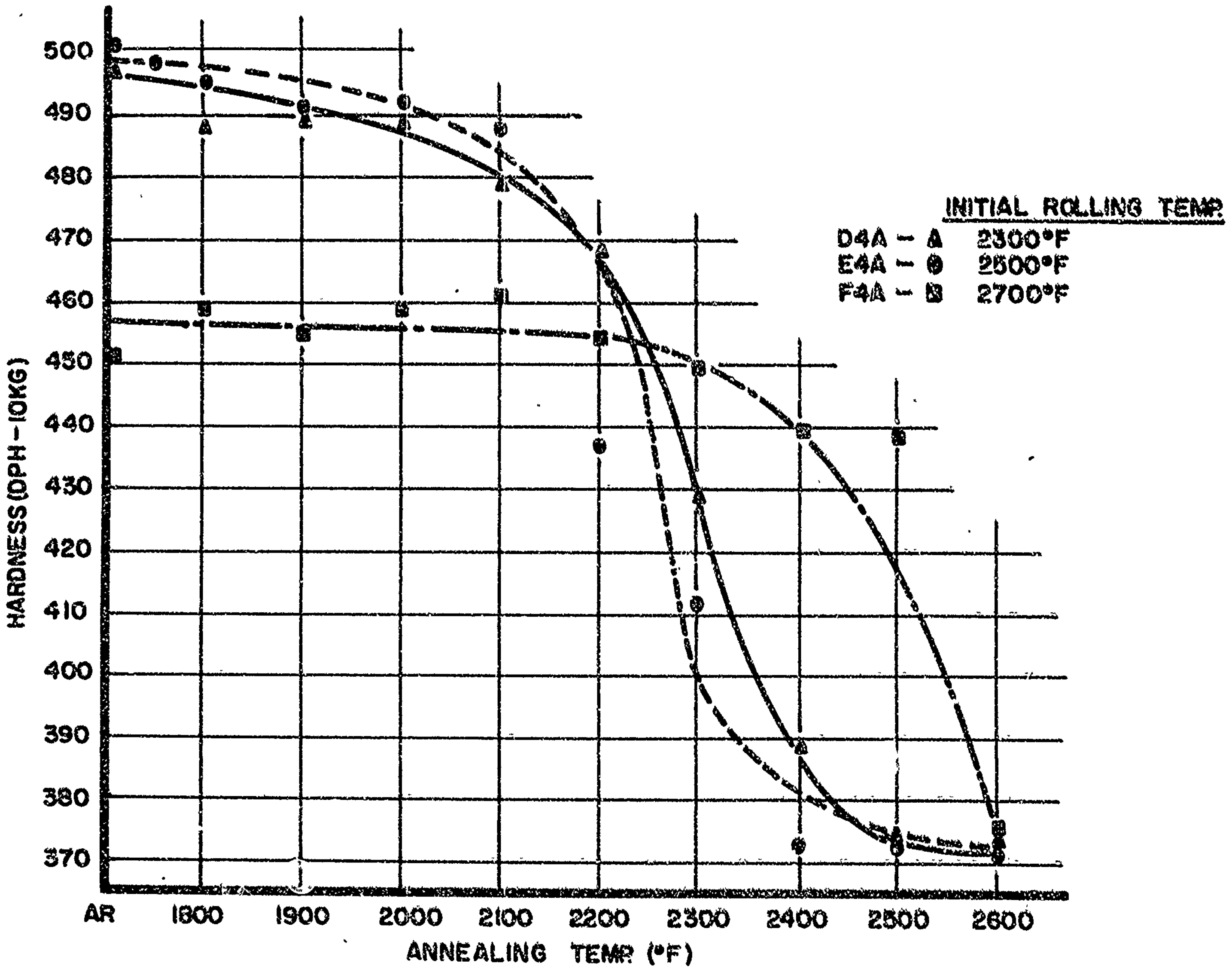


FIGURE IV-14
EFFECT OF INITIAL ROLLING TEMPERATURE ON THE RESPONSE TO HEAT TREATMENT (CONSTANT REDUCTION)

APPENDIX V

MATERIAL ACCOUNTABILITY

I. Introduction

In order to show the amount of material used on this program and the disposition of it, a material control system was established. All material passing through any operation was weighed and thus full accountability was always available.

II. Summary

Table V-I gives a breakdown of all material purchased on this contract. This represents all of the material that was melted for all size ingots employed for this development.

Throughout the program, pieces of material were shipped to various people at the direction of the Air Force. These shipments are summarized in Table V-II.

After all the testing and evaluations were complete, very little sheet remained. Table V-III shows the material on hand at the end of the program.

To summarize the material flow, Table V-IV was compiled and is included to show total disposition. All of the material has been disposed as of this writing.

TABLE V-I

MATERIAL RECEIPTS

KC Electrode Bar

Purchase Order Number	Date Received	Pounds Received
B-3518	5-31-61	296.1
U-11784-2	6-29-61	447.2
U-11784-3	11-29-61	120.6
U-11784-4	4-12-62	920.2
U-11784-5	6-18-62	387.9
		2172.0

KD Electrode Bar

Purchase Order Number	Date Received	Pounds Received
B-11784-1	7-18-61	856.0
B-11784-6	9-11-62	557.0
B-11784-7	9-25-62	698.1
B-11784-8	11-28-62	1299.7
B-11784-9	1-11-63	1544.5
B-11784-11	7-10-63	1793.5
B-11784-12	9-24-63	1799.0
B-11784-13	12-11-63	2947.0
B-17957	1-27-64	5170.0
		16664.8
	Less 575.0 (Trans to RMI-636)	575.0
		16089.8

Miscellaneous

Pad	312.5
Nipple Stock	129.75
	442.25

TABLE V-II

MATERIAL SHIPPED

Heat Number	Pieces	Dimensions	Weight	Customer	Date Shipped
KD1167	1	.060" x 21-1/2" x 13-1/2"	12# 5oz	GE - McGready	5-13-63
KD1147	1	.020" x 34" x 12"	6# 3oz	DuPont - Wartell	5-28-63
KD1167	1	.060" x 25" x 11-1/2"	12# 4oz	TRW - Jeffries	5-28-63
KD1148	2	.020" x 12" x 36"	13# 12oz	DuPont - Wartell	6-6-63
KD1167	1	.060" x 6" x 10-1/8"	2# 11oz	Oak Ridge - Slaughter	6-25-63
KD1168	1	.040" x 9-3/4" x 23-1/2"	6# 11oz	Martin - Schwartzberg	8-26-63
Various	9	.020" x R/W x R/L	19# 7oz	Martin - Schwartzberg	8-26-63
KD1275	8	.060" x 3-1/4" x 3-1/4"	3# 12oz	Kirtland Air Force Base	1-15-65
KD1275	4	.060" x 4" x 6"	4# 4oz	Kirtland Air Force Base	1-15-65
KD1289	6	.080" x 4" x 6"	9#	Kirtland Air Force Base	1-22-65
KD1290	5	.030" x 3" x 8"	2# 8oz	Ford Motor Company	7-14-65
KD1148	3	.040" x 6" x 11-1/16"	5# 3oz	DuPont - Wartell	
KD1289	1	.020" x 12" x 12"	2# 4oz	Kirtland Air Force Base	6-21-66
		Total	100# 4oz		

TABLE V-III

FINISHED MATERIAL IN STOCK

Heat Number	Dimensions	Weight (lbs)	Total Weight (lbs)
KD1274-3C	.022" x 17-7/8" x 55-1/4"	15-1/2	
KD1275-2-1	.030" x 17-1/2" x 35-5/8"	13-1/4	
KD1275-2-2	.040" x 24-1/8" x 72"	54	
KD1275-3B	.058" x 18" x 15-1/2"	11-3/4	
KD1275-3C	.060" x 16-1/2" x 56"	39	
	Development Sheet		133-1/2
KD1287-2	.058" x 12" x 48"	23-1/2	
KD1287-2	.056" x 7" x 23-1/2"	6-1/2	
KD1287-2	.059" x 9-1/4" x 18-3/8"	7	
KD1288-1	.052" x 25-1/2" x 50-3/4"	48	
KD1288-2	.059" x 7-1/2" x 13-3/8"	4	
KD1289-2	.090" x 12-1/2" x 6-1/8"	5	
KD1289-3	.057" x 9-11/16" x 23-5/8"	9	
KD1289-3	.060" x 17-1/4" x 17-3/4"	12-3/4	
KD1289-3	.058" x 10" x 22-1/8"	9	
KD1289-4	.044" x 24" x 72"	52-1/2	
KD1290-1	.063" x 21-1/2" x 38-1/2"	37-1/2	
KD1290-1	.063" x 21-3/8" x 16-1/8"	15-1/2	
KD1290-3	.105" x 6" x 9"	4-1/4	
KD1291-1	.042" x 17-1/2" x 24"	13	
KD1291-1	.043" x 23-3/8" x 12-1/4"	8-3/4	
KD1291-2	.042" x 24" x 30"	21-1/2	
KD1291-2	.042" x 18-1/2" x 21"	11-1/2	
KD1291-3	.040" x 19-5/8" x 23-1/2"	13	
KD1291-3	.041" x 19-1/4" x 21-1/4"	12	
KD1291-4	.045" x 16" x 20"	10-1/4	
KD1291-4	.043" x 10" x 35"	10-3/4	
KD1291-4	.043" x 10-1/2" x 23"	7-1/4	
KD1291-4	.044" x 10-1/2" x 15-1/2"	5	
KD1291-6	.023" x 14-1/4" x 37-1/4"	9	
KD1291-6	.021" x 16" x 20"	5	
	Pilot Production Sheet		361-1/2
	Total Weight		495

TABLE V-IV

MATERIAL SUMMARY

	Received	Shipped	On Hand	Process and Evaluation Loss
KC Melt Stock (Table V-I)	2,172.0			
KD Melt Stock (Table V-I)	16,089.8			
Miscellaneous (Table V-I)	442.25			
Finish Sheet (Table V-II)		100.25		
Scrap Transferred		16,640.0		
Finish Material in Stock (Table V-III)			495.0	
	18,704.05	16,740.25	495.0	1,468.8

UNCLASSIFIED

Security Classification

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13. ABSTRACT A manufacturing process has been developed to produce unalloyed arc-cast tungsten sheet materials. The processing parameters for optimum physical and mechanical properties were investigated from the raw material to the final product. The investigation included powder consolidation to electrode, ingot melting variables, primary breakdown, and a study of the effect of rolling variables on final sheet properties. A scale-up of processing to produce pilot production quantities of .020", .040", and .060" gauge sheet was accomplished, but severe processing and handling problems in all phases of the scale-up prevented the realization of the goal, 36" wide x 96" long sheet. Satisfactory tungsten ingots were melted up to 9-1/2" round. Direct forging to sheet bar and press forging of extruded rounds is not practical due to extra operations and yield loss on conditioning. Satisfactory extrusion of sheet bars and rounds from conditioned billet diameters up to 6" can be accomplished at extrusion ratios of 4.1:1 and 3000° to 3500°F furnace temperatures. The optimum bend transition properties were obtained from material having a minimum 92% reduction from the last recrystallization anneal. The lowest longitudinal bend transition temperature achieved was 200°F.

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Arc-Cast						
Sheet						
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Rolling						
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